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# IMPACT OF COOLING RATE ON THE DURABILITY OF CST GLASSES: A Nonproprietary Summary (U)

T. B. Edwards

J. R. Harbour

R. J. Workman

Westinghouse Savannah River Company Savannah River Technology Center Aiken, SC 29808



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# Impact of Cooling Rate on the Durability of CST Glasses: A Nonproprietary Summary (U)

March 8, 2001

Document Approvals	
T. B. Edwards, Author Statistical Consulting Section	Date
J. R. Harbour, Author Immobilization Technology Section	Date
R. J. Workman, Author Immobilization Technology Section	Date
K. G. Brown, Technical Review Immobilization Technology Section	Date
D. K. Peeler, Technical Review Immobilization Technology Section	Date
R. C. Tuckfield Manager Statistical Consulting Section	Date
R. H. Spires, Manager Immobilization Technology and Business Development Group Immobilization Technology Section Authorized Derivative Classifier	Date
The Following Signatures Signify Satisfactory Completion of	f the FY01 CST Glass Study
H. H. Elder, Acting Manager, HLW Process Engineering	Date
J. T. Carter, Director, HLW Salt Waste Program Engineering	Date
D. W. Wester, TFA CST System Lead, Pacific Northwest National Laboratory	 Date

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# **SUMMARY AND CONCLUSION**

This report is a modified version of WSRC-TR-2001-00124, Revision 0. Information related to the chemical compositions of the study glasses has been removed from this version. WSRC has submitted a patent application for the frit (designated as BD1) utilized in the study and is limiting the distribution of the details of the compositions of the study glasses to protect its intellectual property rights pending patent clearance.

The crystalline silicotitanate (CST) glass study was conducted to determine the effect, if any, on the Product Consistency Test (PCT) responses of CST glasses cooled at different rates. The glasses contained CST and monosodium titanate (MST) [plus a simulated sludge representing Purex, Blend, or HM] in amounts consistent with coupled operations using the CST option for salt disposition. Two bounding cooling profiles were used in this study: rapidly quenched and canister centerline cooled. Glasses were selected based on a number of criteria, but mainly to challenge the regions where amorphous phase separation might be expected. Currently, DWPF utilizes a homogeneity constraint to preclude glass regions that could lead to phase separation. However, this constraint was not developed for a compositional region that includes the levels of CST (3-9 wt%) and MST (2.5 wt%) introduced by this study. Due to this uncertainty, the CST glasses that were selected for study covered a wide range of values for the homogeneity constraint. Also, it was important to ensure that deleterious phase separation does not occur for either cooling profile. In this case, deleterious phase separation is defined as the formation of amorphous phase separation (or glass-in-glass) that significantly alters the glass durability as measured by the PCT response.

The durabilities, as measured by the PCT response of boron, for the CST glasses ranged from 0.41 to 0.80 g/L. These values are significantly lower than the PCT values for glasses containing precipitate hydrolysis aqueous (PHA) [as reported in WSRC-TR-2001-00123] and for environmental assessment (EA). The PCT results are highly clustered consistent with previous work. Many of the measured PCT values fall above the upper 95% prediction interval of the DWPF model used to predict durability, again, consistent with previous results. The very good durability of the CST containing glasses implies that durability may not be the limiting factor for waste loading in this option.

X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to evaluate crystalline phases observed in the centerline cooled glasses. XRD analysis, which detects crystals throughout the bulk of the glass, revealed that trevorite and hematite were present in some of the glasses.

SEM analyses of the crystals that formed on the surface of the glasses revealed a variety of different species including titanium rods and spots, trevorite, trevorite enriched with titanium (which appears to correlate with the amount of titanium in the glass), and a phase rich in Mn and Fe. One glass, cst12, contained a crystal high in uranium content.

The results revealed that there was no practical difference between the PCT responses for glasses subjected to the two cooling profiles. In fact, although to a small extent, the boron PCT responses for the centerline-cooled glasses were more durable than the rapidly quenched glasses. These results reveal that no deleterious amorphous phase separation or crystalline phase occurred under either cooling regime for the glass compositions tested.

# Introduction

One of the Alternative Salt Disposition Flowsheets being considered would require that the Defense Waste Processing Facility (DWPF) vitrify a coupled feed containing High Level Waste (HLW) and crystalline silicotitanate (CST). A Technical Task Request (TTR) [1] was received as part of the Salt Processing Program by the Savannah River Technology Center (SRTC) requesting that a glass variability study be conducted to explore the impact of cooling rate on glass durability for this alternative. A Task Technical and Quality Assurance (TT&QA) plan [2] was issued by SRTC in response to the TTR. The objective of this task is to study the impact of cooling rate on the durability

of glass with anticipated levels of CST in DWPF glasses with nominal levels of monosodium titanate (MST).

The set of target glass compositions for this task was provided in WSRC-TR-2001-00124, Revision 0. The study glasses were selected from a set of candidate glasses that covered Purex, HM, and Blend sludge types at loadings (in the glass) of 22 to 30 oxide weight percent (wt%), utilized CST loadings (in the glass) of 3, 6, and 9 oxide wt%, and included MST concentrations (in the glass) mainly at 2.5 wt%. A  $\pm 10\%$  variation in the composition of each sludge type was also utilized in the development of the set of candidate glass compositions. For each composition, the remainder of the glass consisted of glass formers collectively referred to as BD1 $^1$ , the composition of which is WSRC proprietary information.

Compositions were selected from the set of candidate points that increased the opportunity of seeing any significant cooling impact on glass durability as measured by the PCT. Several glasses were chosen that would also demonstrate reproducibility of the results from an earlier study [3]. The general targeted compositions for the CST glasses are provided in Table 1.

Table 1: Targeted Compositions (in wt%) for the CST FY01 Study

				rtuuj	
Glass	Sludge				BD1
ID	Type	Sludge	MST	CST	Frit
cst01	Purex	26	2.50	9	62.50
cst02	HM	26	1.25	6	66.75
cst03	Purex	22	2.50	9	66.50
cst04	Purex	30	1.25	6	62.75
cst05	Purex	26	2.50	3	68.50
cst06	Purex	30	1.25	9	59.75
cst07	Purex	22	1.25	3	73.75
cst08	Purex	30	2.50	6	61.50
cst09	Purex	26	1.25	9	63.75
cst10	Blend	30	2.50	6	61.50
cst11	Blend	28	2.50	9	60.50
cst12	Blend	30	1.25	3	65.75
cst13	Blend	26	1.25	6	66.75
cst14	HM	26	1.25	6	66.75
cst15	Blend	26	1.25	6	66.75
cst16	HM	26	1.25	6	66.75
cst17	Blend	26	1.25	6	66.75

Property models [4] that are utilized by the DWPF for process and product quality control were used to help in the selection of the study glasses. The process properties of interest included viscosity and liquidus temperature. The product property of interest for the study glasses is durability (as measured by the 7-day Product Consistency Test (PCT) [5]). Currently, DWPF utilizes a homogeneity constraint to preclude glass regions that could lead to amorphous phase separation. However, this constraint was not developed for a compositional region that includes the levels of CST (3-9 wt%) and MST (2.5 wt%) introduced by this study [6]. Due to this uncertainty, the CST glasses that were selected for study covered a wide range of values for the homogeneity constraint. These property predictions along with other key characteristics of the targeted compositions are provided in Table 2.

A patent application has been submitted for the formulation of BD1, and, thus, the details associated with the chemical compositions of the study glasses are not provided in this report.

**Table 2: Property Predictions for the CST Study Glasses** 

										Normalized Releases				
Glass	Sludge		Loa	adings		Al <sub>2</sub> O <sub>3</sub>	alkalis	Homo		В	Li	Na	Visc.	$T_{\rm L}$
ID	Type	S	MST	CST	Frit	(wt%)	(wt%)	(wt%)	del Gp	(g/L)	(g/L)	(g/L)	<b>(P)</b>	°C
cst01	Purex	26	2.50	9	62.50	2.870	13.12	197.9	-6.3390	0.18	0.24	0.19	72.8	994.8
cst02	HM	26	1.25	6	66.75	7.060	13.09	204.9	-5.2664	0.11	0.17	0.13	162.2	926.3
cst03	Purex	22	2.50	9	66.50	2.720	13.43	186.2	-6.6769	0.20	0.27	0.22	89.6	939.8
cst04	Purex	30	1.25	6	62.75	3.550	13.73	205.5	-7.3432	0.27	0.34	0.28	67.9	1007.3
cst05	Purex	26	2.50	3	68.50	3.160	13.99	207.3	-7.3937	0.27	0.35	0.29	73.8	983.1
cst06	Purex	30	1.25	9	59.75	3.530	13.46	209.7	-6.6454	0.20	0.27	0.22	60.7	1038.5
cst07	Purex	22	1.25	3	73.75	2.340	13.49	203.3	-7.2146	0.25	0.33	0.27	88.2	955.7
cst08	Purex	30	2.50	6	61.50	2.960	12.97	214.6	-6.3417	0.18	0.24	0.19	58.5	1054.2
cst09	Purex	26	1.25	9	63.75	2.630	12.83	197.5	-6.4721	0.19	0.25	0.20	78.2	986.7
cst10	Blend	30	2.50	6	61.50	4.440	11.78	208.9	-5.0590	0.10	0.16	0.12	105.1	1009.8
cst11	Blend	28	2.50	9	60.50	4.290	11.28	209.8	-4.1571	0.07	0.12	0.08	103.8	1023.5
cst12	Blend	30	1.25	3	65.75	4.600	11.70	223.3	-5.0887	0.10	0.16	0.12	103.9	1026.8
cst13	Blend	26	1.25	6	66.75	4.320	13.24	206.5	-6.4123	0.18	0.25	0.20	94.1	978.3
cst14	HM	26	1.25	6	66.75	7.060	14.76	204.9	-6.8053	0.21	0.28	0.23	120.9	931.1
cst15	Blend	26	1.25	6	66.75	4.320	13.57	206.5	-6.7172	0.21	0.28	0.22	88.3	979.6
cst16	HM	26	1.25	6	66.75	7.060	15.76	204.9	-7.7293	0.31	0.39	0.33	100.5	934.1
cst17	Blend	26	1.25	6	66.75	4.320	14.57	206.5	-7.6419	0.30	0.38	0.32	72.5	983.6

The purpose of this report is to provide and investigate comparisons between

- the measured and target compositions of the CST glasses,
  - the PCT measurements and durability model predictions, and
- the PCT measurements for quenched versus centerline-cooled glasses.

Evaluating the results from these comparisons provides the basis for assessing the impact of cooling rate on the durability of CST glasses.

# **RESULTS AND DISCUSSION**

The 17 glasses comprising the CST study were designated as cst01 through cst17. Composition and PCT measurements of these glasses were conducted in accordance with analytical plans that were used to generate the measurements supporting the CST study. (See WSRC-TR-2001-00124, Revision 0.) These plans were prepared to support the overall Task Technical and QA plan [2] and the analytical study plan [7]. The results of the composition measurements were provided in WSRC-TR-2001-00124, Revision 0 and are not discussed in this report.

Predictions for the properties of interest that were generated for these target compositions by the models utilized by the DWPF were also included in WSRC-TR-2001-00124, Revision 0. These properties relate a given composition to its processability and product quality. For a given composition, acceptable property characteristics and reliable property predictions (using the current DWPF models) are of interest. The critical property for this study is glass durability. Comparisons between predictions and measurements for this property are provided for both cooling treatments in the discussion that follows.

#### **Glass Batching and Cooling**

Glasses were batched and fabricated to targeted compositions and were identified by cst01 though cst17 as indicated in Table 1. Since the compositions of the simulated sludges were allowed to vary  $\pm 10\%$  around their respective nominal compositions, their contributions to the CST glasses had to be represented as dry chemicals (e.g., reagent grade nitrates, carbonates, and oxides). The contributions of the glass formers (as BD1) to the composition of the study glasses were introduced the same way. The contribution of the MST was represented by appropriate addition of this material in the batching of each glass.

For each glass, the combined powders (~120 grams) were added to a 100 mL Pt-Au crucible and placed in a calibrated furnace that was heated to 1150°C at a rate of 10°C/minute and then held for four hours at 1150°C. Then the crucible was removed and the glass was immediately poured onto a clean stainless steel plate, resulting in what is referred to as a "quenched" glass.

A portion of each glass cooled by quenching was then remelted and allowed to cool following a centerline cooling profile. This was accomplished by taking a portion of each glass and placing it in a smaller crucible. Then, the crucible was introduced to a furnace at 1150°C. The furnace was programmed to cool at a rate that simulated the centerline cooling profile of DWPF canisters.

#### **Chemical Compositions**

In addition to the CST glasses, a standard glass (Batch 1) and a standard uranium-bearing glass were included in the planning of these analyses. These standards were used to correct for potential biases in the measured chemical compositions of the study glasses. Once again, a detailed discussion of the chemical compositions is provided in WSRC-TR-2001-00124, Revision 0. However, in the discussion that follows three options for the compositions of the glasses [targeted, measured, and measured bias corrected (bc)] are provided.

#### **PCT Results**

Samples of the 17 CST glasses, after being batched and fabricated (via quenching), were subjected to a second heat treatment. They were cooled to simulate a canister-centerline-cooling profile. Differences in glass durability for these two cooling regimes (quenched versus centerline cooled) are of primary interest to this study. The investigation into this question required durability to be measured for the quenched and centerline cooled versions of each of the study glasses.

The 7-day Product Consistency Test (PCT) was used as the assessment of glass durability [5]. More specifically, Method A of the PCT (ASTM C1285) was used for these measurements. The PCTs were conducted in triplicate for the CST glasses. In addition, PCTs were also conducted in triplicate for samples of the Environmental Assessment (EA) glass, the Approved Reference Material (ARM) glass, and a blank (ASTM Type I water). An analytical plan (see WSRC-TR-2001-00124, Revision 0) supporting these tests was provided to assist the SRTC-ML in measuring the compositions of the solutions resulting from these PCTs. Of primary interest were the concentrations (in parts per million, ppm) of boron (B), lithium (Li), sodium (Na), and silicon (Si). Samples of a multi-element solution standard were also included in the analytical plan (as a check on the accuracy of the inductively coupled plasma (ICP) – emission spectrometer used for these measurements).

The results from these tests are given in Table A.1 of Appendix A. The PCT results for the centerline-cooled version of each CST study glass are indicated by the "clc" suffix on the glass ID. One of the quality control checkpoints for the PCT procedure is solution-weight loss over the course of the 7-day test. The shaded entries of Table A3 indicate those solutions that fell outside the weight-loss guidelines (weight loss must be less than 5%). Two successful solutions out of the 3 conducted for a glass are required to generate a representative PCT for that glass [5]. Although this criterion is not met for all of the CST study glasses, the results are believed to provide meaningful and representative comparisons for assessing the impact of the cooling regimes.

Any measurement in the "as reported" columns of Table A.3 proceeded by a "<" was below the detection limit for this measurement. The measurement was replaced by one half of the detection limit in the determination of the parts per million (ppm) columns of the table. The values in the ppm columns were also adjusted for the dilution factors by multiplying the "as-reported" values by 1.6667 for the CST and ARM glasses and by 16.6667 for the EA glass. Thus, the concentrations in the ppm columns reflect detection and dilution adjustments.

Exhibit A.1 in the Appendix provides pairs of plots of the leachate concentrations and standards in the analytical sequence reported by the SRTC-ML. For each element of interest, one plot of the pair includes the values from the EA PCTs and the blanks. These values expand the scales of these plots, making it difficult to distinguish among the results of the other analyses. The other plot excludes the EA and blank results.

Exhibit A.2 in the Appendix provides pairs of plots of the leachate concentrations for each type of submitted solution: the standards, the blanks, EA, ARM, and the CST glasses. One plot of the pair includes EA and the blank. The other does not.

PCT leachate concentrations are typically normalized using the cation composition (expressed as a weight percent) in the glass to obtain a grams-per-liter (g/L) leachate concentration. The normalization of the PCTs is usually conducted using the measured compositions of the glasses. This is the preferred normalization process for the PCTs. For completeness, the targeted cation and the bias-corrected cation compositions are also used to conduct this normalization.

As is the usual convention, the common logarithm of the normalized PCT (normalized leachate, NL) for each element of interest are determined and used for comparison. To accomplish this computation, one must

- 1. Determine the common logarithm of the elemental parts per million (ppm) leachate concentration for each of the triplicates and each of the elements of interest (these values are provided in Table A.3 of Appendix A),
- Average the common logarithms over the triplicates for each element of interest, and then

Normalizing Using Measured Composition (preferred method)

3. Subtract a quantity equal to 1 plus the common logarithm of the average cation measured concentration (expressed as a weight percent of the glass) from the average computed in step 2.

#### Or Normalizing Using Target Composition

3. Subtract a quantity equal to 1 plus the common logarithm of the target cation concentration (expressed as a weight percent of the glass) from the average computed in step 2.

# Or Normalizing Using Measured Bias-Corrected Composition

3. Subtract a quantity equal to 1 plus the common logarithm of the measured bias-corrected cation concentration (expressed as a weight percent of the glass) from the average computed in step 2.

As a preliminary step to completing these normalizations of the PCTs, statistical analyses were conducted on the three measurements of the multi-element standard solution per analytical block. Exhibit A.3 in the Appendix provides these analyses. Although there appears to be statistical differences among the block averages, no bias correction of the PCT results for the study glasses was conducted. This approach was taken since the triplicate PCTs for a single study glass were placed in different ICP blocks. Averaging the ppm's for each set of triplicates helps to minimize the impact of the ICP effects.

The block averages are presented in Table 3. They indicate consistent and reasonably accurate results (differences of overall averages versus reference values < 5%) from these analyses.

**Table 3: Measurements of Standard Solution** 

Analytical	Avg	Avg	Avg	Avg
Block	B (ppm)	Li (ppm)	Na (ppm)	Si (ppm)
1	19.6	9.5	82.6	49.7
2	19.3	9.3	77.8	49.8
3	20.1	9.9	78.6	50.2
4	19.6	10.4	80.5	48.5
5	19.3	9.5	81.4	50.1
6	20.0	10.1	80.7	51.2
Grand Average	19.6	9.8	80.3	49.9
Reference Value	20	10	81	50
% difference	-1.8%	-2.2%	-0.9%	-0.2%

Table 4 provides the results from the normalization process using the information on the chemical compositions of the glasses (see WSRC-TR-2001-00124, Revision 0) and all of the data of Table A.1 (i.e., before screening the PCT results for solution weight problems). Exhibit A.4 in Appendix A provides scatter plots for these results (both quenched and centerline-cooled) and offers an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. The consistency is typically demonstrated by a high degree of linear correlation among the values. PCT values normalized using targeted, measured, and bias-corrected compositions are investigated. A high degree of correlation is not seen for these data for many pairs of the elements. The largest correlation (~81%) is between B and Li for the unscreened PCTs normalized using the targeted compositions. The smallest correlation (~38%) is between Li and Na. These poor correlations may be due in part to the relative insensitivity of the PCT responses to the compositional variations of the study glasses. For example, all of the log NL[B (g/L)] values of Table 4 fall within the interval (-0.382, -0.099) for the CST study glasses.

Table 4: Normalized PCTs before Screening for Solution-Weight Problems

		Quenched							Centerline Cooled								
Glass		loo NI	loo NII	loo NII	log NL	NL	NL	NL	NL	log NL	log NL	log NL	log NL	NL	NL	NL	NL
ID	Composition	log NL [B(g/L)]	log NL	log NL						_	_	_	_				
ARM	see [8]	-0.2175	[Li(g/L)] -0.1726	[Na(g/L)] -0.2427	[Si(g/L)] -0.5081	B(g/L) 0.61	Li(g/L) 0.67	Na(g/L) 0.57	Si(g/L) 0.31	[B(g/L)]	[Li(g/L)]	[Na(g/L)]	[Si(g/L)]	D(g/L)	LI(g/L)	Na(g/L)	31(g/L)
EA	see [8]	0.9128	0.7206	0.8196	0.3597	8.18	5.26	6.60	2.29								
LA	Meas.	-0.1842	-0.0724	-0.3483	-0.3823	0.65	0.85	0.45	0.41	-0.2276	-0.1233	-0.3668	-0.3718	0.59	0.75	0.43	0.42
cst01	Meas. bc	-0.1842	-0.0724	-0.3434	-0.3786	0.66	0.83	0.45	0.41	-0.2223	-0.1233	-0.3619	-0.3681	0.60	0.73	0.43	0.42
CStO1	Target	-0.1733	-0.1042	-0.3388	-0.3693	0.63	0.79	0.46	0.43	-0.2408	-0.1550	-0.3573	-0.3588	0.57	0.70	0.44	0.44
	Meas.	-0.3771	-0.1144	-0.6999	-0.4168	0.42	0.77	0.20	0.38	-0.3764	-0.1477	-0.6499	-0.4121	0.42	0.71	0.22	0.39
cst02	Meas. bc	-0.3718	-0.1304	-0.6950	-0.4131	0.42	0.74	0.20	0.39	-0.3712	-0.1637	-0.6450	-0.4084	0.43	0.69	0.23	0.39
	Target	-0.3820	-0.1355	-0.6752	-0.4092	0.41	0.73	0.21	0.39	-0.3813	-0.1688	-0.6253	-0.4044	0.42	0.68	0.24	0.39
	Meas.	-0.2267	-0.0846	-0.3606	-0.3569	0.59	0.82	0.44	0.44	-0.2239	-0.1099	-0.3509	-0.3402	0.60	0.78	0.45	0.46
cst03	Meas. bc	-0.2214	-0.1006	-0.3558	-0.3531	0.60	0.79	0.44	0.44	-0.2186	-0.1259	-0.3461	-0.3364	0.60	0.75	0.45	0.46
	Target	-0.2309	-0.1066	-0.3615	-0.3566	0.59	0.78	0.43	0.44	-0.2281	-0.1319	-0.3518	-0.3399	0.59	0.74	0.44	0.46
	Meas.	-0.1777	-0.0599	-0.2814	-0.3587	0.66	0.87	0.52	0.44	-0.2117	-0.0987	-0.2930	-0.3638	0.61	0.80	0.51	0.43
cst04	Meas. bc	-0.1724	-0.0758	-0.2803	-0.3573	0.67	0.84	0.52	0.44	-0.2064	-0.1146	-0.2918	-0.3624	0.62	0.77	0.51	0.43
	Target	-0.1780	-0.0798	-0.2989	-0.3480	0.66	0.83	0.50	0.45	-0.2120	-0.1186	-0.3105	-0.3531	0.61	0.76	0.49	0.44
	Meas.	-0.1752	-0.0636	-0.2826	-0.3578	0.67	0.86	0.52	0.44	-0.1742	-0.0906	-0.2655	-0.3458	0.67	0.81	0.54	0.45
cst05	Meas. bc	-0.1684	-0.0773	-0.2815	-0.3564	0.68	0.84	0.52	0.44	-0.1674	-0.1043	-0.2644	-0.3444	0.68	0.79	0.54	0.45
	Target	-0.1883	-0.0900	-0.2829	-0.3364	0.65	0.81	0.52	0.46	-0.1872	-0.1170	-0.2658	-0.3244	0.65	0.76	0.54	0.47
	Meas.	-0.2090	-0.0686	-0.3183	-0.3959	0.62	0.85	0.48	0.40	-0.3008	-0.1406	-0.3498	-0.3957	0.50	0.72	0.45	0.40
cst06	Meas. bc	-0.2023	-0.0824	-0.3135	-0.3922	0.63	0.83	0.49	0.41	-0.2940	-0.1544	-0.3450	-0.3920	0.51	0.70	0.45	0.41
	Target	-0.2009	-0.0868	-0.3194	-0.3846	0.63	0.82	0.48	0.41	-0.2926	-0.1588	-0.3509	-0.3844	0.51	0.69	0.45	0.41
	Meas.	-0.1048	-0.0135	-0.3020	-0.2840	0.79	0.97	0.50	0.52	-0.1345	-0.0555	-0.2991	-0.2944	0.73	0.88	0.50	0.51
cst07	Meas. bc	-0.0994	-0.0294	-0.2971	-0.2803	0.80	0.93	0.50	0.52	-0.1291	-0.0714	-0.2942	-0.2907	0.74	0.85	0.51	0.51
	Target	-0.1244	-0.0401	-0.2704	-0.2897	0.75	0.91	0.54	0.51	-0.1541	-0.0822	-0.2675	-0.3001	0.70	0.83	0.54	0.50
	Meas.	-0.1369	-0.0495	-0.2837	-0.3506	0.73	0.89	0.52	0.45	-0.2490	-0.1318	-0.3286	-0.3701	0.56	0.74	0.47	0.43
cst08	Meas. bc	-0.1300	-0.0632	-0.2788	-0.3469	0.74	0.86	0.53	0.45	-0.2422	-0.1455	-0.3237	-0.3664	0.57	0.72	0.47	0.43
	Target	-0.1344	-0.0625	-0.2627	-0.3313	0.73	0.87	0.55	0.47	-0.2466	-0.1448	-0.3077	-0.3509	0.57	0.72	0.49	0.45
	Meas.	-0.1669	-0.0325	-0.3179	-0.3680	0.68	0.93	0.48	0.43	-0.2109	-0.0928	-0.3397	-0.3902	0.62	0.81	0.46	0.41
cst09	Meas. bc	-0.1615	-0.0484	-0.3168	-0.3666	0.69	0.89	0.48	0.43	-0.2055	-0.1087	-0.3386	-0.3888	0.62	0.78	0.46	0.41
	Target	-0.1800	-0.0653	-0.3307	-0.3515	0.66	0.86	0.47	0.45	-0.2240	-0.1256	-0.3525	-0.3737	0.60	0.75	0.44	0.42
	Meas.	-0.2618	-0.0932	-0.5127	-0.4115	0.55	0.81	0.31	0.39	-0.3545	-0.1720	-0.5228	-0.4307	0.44	0.67	0.30	0.37
cst10	Meas. bc	-0.2550	-0.1070	-0.5115	-0.4101	0.56	0.78	0.31	0.39	-0.3477	-0.1858	-0.5217	-0.4293	0.45	0.65	0.30	0.37
	Target	-0.2635	-0.1155	-0.5055	-0.3966	0.55	0.77	0.31	0.40	-0.3562	-0.1943	-0.5156	-0.4158	0.44	0.64	0.31	0.38
	Meas.	-0.2675	-0.0817	-0.5961	-0.4133	0.54	0.83	0.25	0.39	-0.3479	-0.1309	-0.5578	-0.4301	0.45	0.74	0.28	0.37
cst11	Meas. bc	-0.2607	-0.0955	-0.5913	-0.4096	0.55	0.80	0.26	0.39	-0.3411	-0.1446	-0.5530	-0.4264	0.46	0.72	0.28	0.37
	Target	-0.2751	-0.1055	-0.5922	-0.4054	0.53	0.78	0.26	0.39	-0.3555	-0.1547	-0.5539	-0.4222	0.44	0.70	0.28	0.38
	Meas.	-0.2313	-0.0754	-0.4997	-0.3840	0.59	0.84	0.32	0.41	-0.2677	-0.1314	-0.4670	-0.4060	0.54	0.74	0.34	0.39
cst12	Meas. bc	-0.2260	-0.0914	-0.4985	-0.3827	0.59	0.81	0.32	0.41	-0.2623	-0.1474	-0.4658	-0.4046	0.55	0.71	0.34	0.39
	Target	-0.2373	-0.1022	-0.5034	-0.3713	0.58	0.79	0.31	0.43	-0.2737	-0.1582	-0.4707	-0.3933	0.53	0.69	0.34	0.40
cst13	Meas. Meas. bc	-0.2660 -0.2592	-0.0898 -0.1035	-0.4529 -0.4519	-0.4035 -0.4021	0.54 0.55	0.81 0.79	0.35 0.35	0.39 0.40	-0.2638 -0.2570	-0.1078 -0.1215	-0.4083 -0.4072	-0.3921 -0.3907	0.54	0.78 0.76	0.39	0.41 0.41
CSU13														0.55			
<b>-</b>	Target	-0.2727	-0.1155	-0.4397	-0.3896	0.53	0.77	0.36	0.41	-0.2705	-0.1335	-0.3950	-0.3782	0.54	0.74	0.40	0.42
act 1.4	Meas.	-0.3422 -0.3354	-0.1077 -0.1215	-0.4491 -0.4479	-0.3780 -0.3766	0.45	0.78 0.76	0.36 0.36	0.42	-0.3377 -0.3310	-0.1233 -0.1371	-0.4137 -0.4125	-0.3739	0.46	0.75	0.39	0.42 0.42
cst14	Meas. bc					0.46	0.76		0.42				-0.3725	0.47	0.73	0.39	
	Target Meas.	-0.3432 -0.2454	-0.1358 -0.0765	-0.4457 -0.4023	-0.3730 -0.3903	0.45	0.73	0.36	0.42	-0.3388 -0.2560	-0.1514 -0.1100	-0.4103 -0.3775	-0.3689 -0.3862	0.46	0.71	0.39	0.43
cst15	Meas. bc	-0.2454	-0.0763	-0.4023	-0.3889	0.57	0.84	0.40	0.41	-0.2360	-0.1100	-0.3775 -0.3764	-0.3862	0.55	0.78	0.42	0.41
CSLID	Target	-0.2546	-0.0903	-0.4012	-0.3889	0.58	0.81	0.40	0.41	-0.2492	-0.1238	-0.3764 -0.3781	-0.3849	0.56	0.73	0.42	0.41
	Meas.	-0.3114	-0.1495	-0.3566	-0.3772	0.49	0.71	0.44	0.42	-0.2725	-0.1361	-0.3410	-0.3652	0.53	0.73	0.42	0.42
cst16	Meas. bc	-0.3114	-0.1495	-0.3556	-0.3772	0.49	0.71	0.44	0.42	-0.2725	-0.1460	-0.3410	-0.3632	0.53	0.71	0.46	0.43
CSLIU	Target	-0.3239	-0.1700	-0.3534	-0.3687	0.30	0.68	0.44	0.42	-0.2850	-0.1398	-0.3398	-0.3568	0.54	0.68	0.46	0.43
	Meas.	-0.2150	-0.1700	-0.2995	-0.3540	0.47	0.80	0.50	0.43	-0.2589	-0.1461	-0.3423	-0.3634	0.55	0.08	0.49	0.43
cst17	Meas. bc	-0.2130	-0.0937	-0.2993	-0.3503	0.61	0.80	0.50	0.44	-0.2535	-0.1461	-0.3073	-0.3634	0.56	0.71	0.49	0.43
CSLIT	Target	-0.2193	-0.1113	-0.2979	-0.3428	0.60	0.77	0.51	0.45	-0.2632	-0.1632	-0.3027	-0.3523	0.55	0.69	0.30	0.44
								2.50	20		002		2.2020				

Table 5 provides the results from the normalization process using the information on the chemical compositions of the glasses and the screened data of Table A.1 (i.e., after screening the PCT results for solution weight problems). A valid PCT [5] for a glass requires that at least 2 of the 3 triplicates pass the solution-weight criterion (a weight loss of less than 5%). The glasses whose PCT results do not satisfy that criterion are shaded in Table 5. All of the other entries of Table 5 are deemed valid PCTs. Exhibit A.5 in Appendix A provides correlations and scatter plots for the screened results (including both quenched and centerline-cooled PCTs) and offers an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. Correlations and scatter plots of just the valid PCTs are also provided as part of this exhibit. The consistency is typically demonstrated by a high degree of linear correlation among the values. PCT normalized using targeted, measured, and bias-corrected compositions are investigated. A high degree of correlation is not seen for these data for many pairs of the elements. The largest correlation is only ~82%; the smallest, only slightly over 27%.

Table 5: Normalized PCTs after Screening for Solution-Weight Problems<sup>2</sup>

		14	ibic 5.	1 101 1116	mzcu i	CIS	inng i	ng for Solution-Weight Problems <sup>2</sup>									
	ļ				Quench								enterline Co				
Glass		log NL	log NL	log NL	log NL	NL	NL	NL	NL	log NL	log NL	log NL	log NL	NL	NL	NL	NL
ID	Composition			[Na(g/L)]		B(g/L)	Si(g/L)			[B(g/L)]	[Si(g/L)]	[Na(g/L)]	[Li(g/L)]	B(g/L)	Si(g/L)	Na(g/L)	Li(g/L)
ARM	see [8]	-0.2175	-0.1726	-0.2427	-0.5081	0.61	0.67	0.57	0.31								
EA	see [8]	0.9128	0.7206	0.8196	0.3597	8.18	5.26	6.60	2.29								
01	Meas.	-0.1915	-0.0747	-0.3547	-0.3912	0.64	0.84	0.44	0.41	-0.2276	-0.1233	-0.3668	-0.3718	0.59	0.75	0.43	0.42
cst01	Meas. bc	-0.1862	-0.0907	-0.3498	-0.3874	0.65	0.81	0.45	0.41	-0.2223	-0.1392	-0.3619	-0.3681	0.60	0.73	0.43	0.43
	Target	-0.2047	-0.1065 -0.1144	-0.3452	-0.3781	0.62	0.78	0.45	0.42	-0.2408	-0.1550 -0.1028	-0.3573	-0.3588	0.57	0.70	0.44	0.44
cst02	Meas. Meas. bc	-0.3771 -0.3718	-0.1144	-0.6999 -0.6950	-0.4168 -0.4131	0.42 0.42	0.77 0.74	0.20 0.20	0.38	-0.3335 -0.3283	-0.1028	-0.5648 -0.5599	-0.4080 -0.4042	0.46 0.47	0.79	0.27	0.39
CS102	Target	-0.3718	-0.1355	-0.6752	-0.4131	0.42	0.74	0.20	0.39	-0.3384	-0.1137	-0.5402	-0.4003	0.46	0.75	0.28	0.39
	Meas.	-0.2472	-0.0996	-0.3542	-0.3730	0.57	0.79	0.44	0.42	-0.2484	-0.1196	-0.3509	-0.3540	0.56	0.76	0.45	0.44
cst03	Meas. bc	-0.2419	-0.1156	-0.3494	-0.3693	0.57	0.77	0.45	0.43	-0.2431	-0.1355	-0.3461	-0.3503	0.57	0.73	0.45	0.45
	Target	-0.2514	-0.1216	-0.3551	-0.3728	0.56	0.76	0.44	0.42	-0.2525	-0.1416	-0.3518	-0.3537	0.56	0.72	0.44	0.44
	Meas.									-0.2117	-0.0987	-0.2930	-0.3638	0.61	0.80	0.51	0.43
cst04	Meas. bc									-0.2064	-0.1146	-0.2918	-0.3624	0.62	0.77	0.51	0.43
	Target									-0.2120	-0.1186	-0.3105	-0.3531	0.61	0.76	0.49	0.44
	Meas.	-0.1682	-0.0546	-0.2785	-0.3645	0.68	0.88	0.53	0.43	-0.1742	-0.0906	-0.2655	-0.3458	0.67	0.81	0.54	0.45
cst05	Meas. bc	-0.1614	-0.0683	-0.2773	-0.3631	0.69	0.85	0.53	0.43	-0.1674	-0.1043	-0.2644	-0.3444	0.68	0.79	0.54	0.45
	Target	-0.1812	-0.0810	-0.2787	-0.3431	0.66	0.83	0.53	0.45	-0.1872	-0.1170	-0.2658	-0.3244	0.65	0.76	0.54	0.47
	Meas.	-0.2309	-0.1023	-0.3551	-0.3946	0.59	0.79	0.44	0.40	-0.3008	-0.1406	-0.3498	-0.3957	0.50	0.72	0.45	0.40
cst06	Meas. bc	-0.2242	-0.1161	-0.3503	-0.3908	0.60	0.77	0.45	0.41	-0.2940	-0.1544	-0.3450	-0.3920	0.51	0.70	0.45	0.41
	Target	-0.2228	-0.1205	-0.3563	-0.3832	0.60	0.76	0.44	0.41	-0.2926	-0.1588	-0.3509	-0.3844	0.51	0.69	0.45	0.41
!	Meas.	-0.1103	-0.0262	-0.3164	-0.2822	0.78	0.94	0.48	0.52	-0.1345	-0.0555	-0.2991	-0.2944	0.73	0.88	0.50	0.51
cst07	Meas. bc	-0.1050	-0.0422	-0.3115 -0.2849	-0.2785	0.79	0.91	0.49	0.53	-0.1291	-0.0714	-0.2942	-0.2907	0.74	0.85	0.51	0.51
	Target	-0.1299	-0.0529		-0.2879	0.74	0.89	0.52	0.52	-0.1541	-0.0822	-0.2675	-0.3001	0.70	0.83	0.54	0.50
cst08	Meas. Meas. bc	-0.1332 -0.1263	-0.0466 -0.0603	-0.2891 -0.2842	-0.3576 -0.3539	0.74 0.75	0.90 0.87	0.51 0.52	0.44 0.44	-0.2603 -0.2534	-0.1444 -0.1581	-0.3324 -0.3275	-0.3732 -0.3694	0.55 0.56	0.72	0.47 0.47	0.42 0.43
CSIUO	Target	-0.1203	-0.0596	-0.2682	-0.3339	0.73	0.87	0.54	0.44	-0.2578	-0.1575	-0.3273	-0.3539	0.55	0.70	0.47	0.43
	Meas.	-0.1669	-0.0325	-0.2082	-0.3680	0.74	0.87	0.48	0.43	-0.1978	-0.1373	-0.3114	-0.3359	0.63	0.70	0.49	0.44
cst09	Meas. bc	-0.1615	-0.0323	-0.3179	-0.3666	0.69	0.93	0.48	0.43	-0.1978	-0.0739	-0.3114	-0.3832	0.64	0.84	0.49	0.41
CSCO	Target	-0.1800	-0.0653	-0.3307	-0.3515	0.66	0.86	0.47	0.45	-0.2109	-0.1067	-0.3242	-0.3687	0.62	0.78	0.47	0.43
	Meas.	-0.2593	-0.0930	-0.5113	-0.4143	0.55	0.81	0.31	0.39	-0.3840	-0.1977	-0.5378	-0.4475	0.41	0.63	0.29	0.36
cst10	Meas. bc	-0.2525	-0.1068	-0.5102	-0.4129	0.56	0.78	0.31	0.39	-0.3772	-0.2115	-0.5366	-0.4461	0.42	0.61	0.29	0.36
	Target	-0.2610	-0.1153	-0.5041	-0.3994	0.55	0.77	0.31	0.40	-0.3856	-0.2200	-0.5306	-0.4326	0.41	0.60	0.29	0.37
	Meas.	-0.2634	-0.0790	-0.5885	-0.4131	0.55	0.83	0.26	0.39	-0.3479	-0.1309	-0.5578	-0.4301	0.45	0.74	0.28	0.37
cst11	Meas. bc	-0.2566	-0.0928	-0.5837	-0.4093	0.55	0.81	0.26	0.39	-0.3411	-0.1446	-0.5530	-0.4264	0.46	0.72	0.28	0.37
	Target	-0.2710	-0.1028	-0.5846	-0.4051	0.54	0.79	0.26	0.39	-0.3555	-0.1547	-0.5539	-0.4222	0.44	0.70	0.28	0.38
	Meas.	-0.2283	-0.0724	-0.4942	-0.3828	0.59	0.85	0.32	0.41	-0.2198	-0.0938	-0.4369	-0.3741	0.60	0.81	0.37	0.42
cst12	Meas. bc	-0.2230	-0.0884	-0.4930	-0.3814	0.60	0.82	0.32	0.42	-0.2145	-0.1098	-0.4357	-0.3727	0.61	0.78	0.37	0.42
	Target	-0.2343	-0.0992	-0.4979	-0.3700	0.58	0.80	0.32	0.43	-0.2259	-0.1206	-0.4406	-0.3613	0.59	0.76	0.36	0.44
	Meas.	-0.2630	-0.0877	-0.4364	-0.3892	0.55	0.82	0.37	0.41	-0.2676	-0.1116	-0.4193	-0.3967	0.54	0.77	0.38	0.40
cst13	Meas. bc	-0.2562	-0.1014	-0.4353	-0.3878	0.55	0.79	0.37	0.41	-0.2609	-0.1254	-0.4182	-0.3953	0.55	0.75	0.38	0.40
	Target	-0.2697	-0.1134	-0.4231	-0.3752	0.54	0.77	0.38	0.42	-0.2744	-0.1373	-0.4060	-0.3828	0.53	0.73	0.39	0.41
<u> </u>	Meas.	-0.3533	-0.1231	-0.4673	-0.3726	0.44	0.75	0.34	0.42	-0.3759	-0.1652	-0.4560	-0.3861	0.42	0.68	0.35	0.41
cst14	Meas. bc	-0.3465	-0.1369	-0.4661	-0.3712	0.45	0.73	0.34	0.43	-0.3691	-0.1790	-0.4548	-0.3847	0.43	0.66	0.35	0.41
	Target	-0.3544	-0.1512	-0.4639	-0.3676	0.44	0.71	0.34	0.43	-0.3770	-0.1932	-0.4526	-0.3811	0.42	0.64	0.35	0.42

Shaded rows in this table indicate glasses whose PCTs would not be considered valid due to less than 2 good results out of the 3 triplicates conducted for that glass. Missing values indicate that none of the three PCTs for the indicated glass were considered valid.

Table 5: Normalized PCTs after Screening for Solution-Weight Problems (continued)

					Quench	ed				Centerline Cooled							
Glass		log NL	log NL	log NL	log NL	NL	NL	NL	NL	log NL	log NL	log NL	log NL	NL	NL	NL	NL
ID	Composition	[B(g/L)]	[Si(g/L)]	[Na(g/L)]	[Li(g/L)]	B(g/L)	Si(g/L)	Na(g/L)	Li(g/L)	[B(g/L)]	[Si(g/L)]	[Na(g/L)]	[Li(g/L)]	B(g/L)	Si(g/L)	Na(g/L)	Li(g/L)
	Meas.	-0.2433	-0.0849	-0.4144	-0.3717	0.57	0.82	0.39	0.42	-0.2745	-0.1296	-0.3969	-0.3921	0.53	0.74	0.40	0.41
cst15	Meas. bc	-0.2365	-0.0986	-0.4133	-0.3703	0.58	0.80	0.39	0.43	-0.2678	-0.1434	-0.3957	-0.3907	0.54	0.72	0.40	0.41
	Target	-0.2524	-0.1129	-0.4149	-0.3598	0.56	0.77	0.38	0.44	-0.2837	-0.1576	-0.3974	-0.3802	0.52	0.70	0.40	0.42
	Meas.	-0.3114	-0.1495	-0.3566	-0.3772	0.49	0.71	0.44	0.42	-0.3067	-0.1652	-0.3372	-0.3600	0.49	0.68	0.46	0.44
cst16	Meas. bc	-0.3045	-0.1632	-0.3554	-0.3758	0.50	0.69	0.44	0.42	-0.2998	-0.1790	-0.3360	-0.3586	0.50	0.66	0.46	0.44
	Target	-0.3239	-0.1700	-0.3581	-0.3687	0.47	0.68	0.44	0.43	-0.3192	-0.1858	-0.3387	-0.3516	0.48	0.65	0.46	0.45
	Meas.	-0.2150	-0.0957	-0.2995	-0.3540	0.61	0.80	0.50	0.44	-0.2589	-0.1461	-0.3075	-0.3634	0.55	0.71	0.49	0.43
cst17	Meas. bc	-0.2096	-0.1115	-0.2946	-0.3503	0.62	0.77	0.51	0.45	-0.2535	-0.1620	-0.3027	-0.3597	0.56	0.69	0.50	0.44
	Target	-0.2193	-0.1128	-0.2979	-0.3428	0.60	0.77	0.50	0.45	-0.2632	-0.1632	-0.3059	-0.3523	0.55	0.69	0.49	0.44

Also, note that the EA PCT response from Tables 4 and 5 is lower than that typically seen for this standard glass [8]. However, the ARM results are at the upper end of their historical values as reported in [8].

As seen in Tables 4 and 5, the durabilities for the CST glasses are much better than those of EA. (This is indicated for each glass by its normalized leachate being much smaller than that of EA.). Figures 1-3 provide an opportunity for a closer look at just the valid PCTs using measured, bias-corrected, and targeted compositions. Each of these figures is a plot of the DWPF model that relates the logarithm of the normalized PCT (in this case for B) to a linear function of a free energy of hydration term ( $\Delta G_n$ , kcal/100g glass) derived from the glass (measured, bias-corrected, or targeted) compositions [8]. Prediction limits (at 95% confidence) for individual PCT results are also plotted around this linear fit. The PCT results for EA (shown as a diamond at  $\Delta G_p \sim -15.5$ ), ARM (shown as a diamond at  $\Delta G_p$  slightly above -10), and the CST glasses (quenched shown as small, closed squares and centerline cooled shown as open circles) are presented on this plot. Note that the CST results reveal acceptable PCTs and that the PCT predictions by the current DWPF durability model for boron mimic the behavior seen in [3]. Specifically, at the more positive  $\Delta G_p$  values for the CST glasses, the model tends to predict durabilities that are lower (more durable) than measured. Exhibit A.6 in Appendix A repeats the plots of Figures 1-3, provides the same plots for Li, Na, and Si for the valid PCTs and provides similar plots for B, Li, Na, and Si for the unscreened and screened PCTs. The behavior seen in these plots for B, Li, Na, and Si is similar to that demonstrated by the B results in Figures 1-3: acceptable but less than ideally predictable durabilities. For these plots, the Na behavior reveals more of a slope in the PCT responses than the other elements.

Figure 1.

log NL[B (g/L)] By del Gp
(Valid PCTs & Measured Compositions)

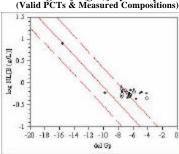


Figure 2.

log NL[B (g/L)] By del Gp
(Valid PCTs & Bias-Corrected Compositions)

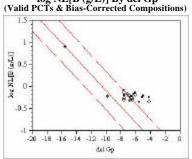
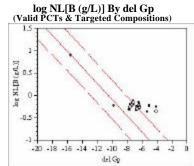


Figure 3.



A look at the reproducibility of the PCT results between this and the FY99 study is provided in Table 6. This table provides the normalized PCT results (in grams/liter) from [3] for the FY99 glasses of interest and from Tables 4 and 5 for the quenched versions of those glasses repeated in this study. Recall that only quenched glasses were studied in the FY99 work. Good reproducibility between the two studies is demonstrated by the results presented in Table 6.

Table 6: Normalized PCTs for Glasses Common to the FY01 and FY99 Studies

Study &	Composition	NL	NL	NL	NL	Glass		NL	NL	NL	NL
Glass ID	Represented By	B(g/L)	Li(g/L)	Na(g/L)	Si(g/L)	ID	Composition	B(g/L)	Li(g/L)	Na(g/L)	Si(g/L)
	Measured	0.64	0.84	0.44	0.41		Measured	0.42	0.77	0.20	0.38
FY01 cst01	Measured bias-cor.	0.65	0.81	0.45	0.41	FY01 cst02	Measured bias-cor.	0.42	0.74	0.20	0.39
	Targeted	0.62	0.78	0.45	0.42		Targeted	0.41	0.73	0.21	0.39
	Measured	0.72	0.86	0.51	0.44		Measured	0.41	0.70	0.25	0.38
FY99 cst12c	Measured bias-cor.	0.72	0.89	0.53	0.44	FY99 cst26	Measured bias-cor.	0.41	0.73	0.25	0.37
	Targeted	0.68	0.86	0.52	0.43		Targeted	0.42	0.71	0.25	0.37

# **Quenched versus Centerline-Cooled PCTs**

A primary objective of this study was the investigation of potential cooling effects on the durability of the CST glasses. Figures 1-3 and Exhibit A.6 provide a first look at differences in PCT response due to the cooling regime for the glasses. From those plots, there does not appear to be a lack of PCT predictability or acceptability due to cooling regime. A more rigorous comparison of the PCTs from quenched versus centerline-cooled glasses is provided in this section.

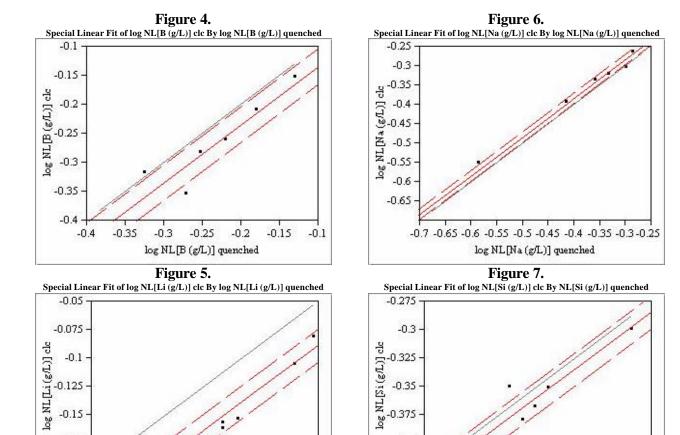
Exhibit A.7 in Appendix A provides paired comparisons (made using JMP® Version 4.3) of the average common logarithm of the leachate concentrations in parts per million. The data that are averaged are provided in Table A.1 of Appendix A. The exhibit investigates the unscreened, screened, and valid PCTs in turn. For boron (regardless of the PCT group), there is a statistically significant difference (at the 5% significance level) between the quenched and centerline-cooled results with the boron PCT releases from the quenched glasses being slightly higher than those from the centerline-cooled glasses. In log space, the difference (on average) is less than 0.03, which is of no real, practical concern. The lithium results mirror those for boron, with the average difference being less than 0.05 in log space and statistically significant at the 5% level.

The results for silicon indicate no statistically significant (at the 5% level) difference in the PCTs due to the heat treatment of the glasses. The mean difference is less than 0.01 in log space for these measurements.

The results for sodium indicate a statistically significant difference (at the 5% level) in the PCTs for only the valid PCTs. In log space, the quenched PCTs are slightly (~0.02) smaller than the centerline-cooled PCTs. Thus, the behavior of the Na response is somewhat different from the behaviors demonstrated for the other elements.

Another look at quenched versus centerline-cooled PCTs for boron is provided in Figures 4-7 for the valid PCTs. These plots provide quenched versus centerline cooled scatter plots with the x and y axes drawn to the same scale. If the PCT is not affected by cooling regime, the quenched and centerline-cooled pairs should fall along the diagonal line for each of these plots.

JMP® Version 4.0 is a commercial software product of SAS Institute, Inc. in Cary, NC. This product was used to support the statistical analyses presented in this report. See JMP® Statistical Discovery Software: Statistics and Graphics Guide (2000) published by SAS Institute for details.



These results indicate no impact (of practical importance) between the two cooling regimes (quenched and centerline cooled) for the CST glasses of this study.

-0.075 -0.05

-0.4

-0.425

-0.4

-0.375 -0.35

log NL[Si (g/L)] quenched

-0.325

-0.3 -0.275

-0.425

### **Phase Separation**

-0.175

-0.15

-0.125

log NL[Li (g/L)] quenched

-0.1

-0.175

-0.2

-0.2

The formation of separate amorphous phases in glass is referred to as amorphous phase separation or inhomogeneity. Crystal formation, as determined by liquidus temperature measurements on the other hand, may indicate a "separation of phases," but reflects crystalline particles within the glass matrix. Amorphous phase separation is to be avoided since the models currently used to predict durability do not apply for glasses predicted to be phase separated. The limit for the homogeneity constraint in the PCCS is nominally (for the Property Acceptance Region, PAR) a value of ~211 [4]. For the measurement acceptance region (MAR), the value will be even higher. In order for a glass to pass this constraint, the calculated value from chemical composition of the glass must be greater than the MAR value. The predicted values for the CST glasses are given in Table 7. All of the targeted compositions except for cst08 are below the PAR and are, thus, predicted to be phase separated. For the measured compositions, only cst06, cst08, and cst12 pass the PAR. For the bias-corrected compositions, only cst08 and cst12 fall within the PAR.

**Table 7: Homogeneity Property Predictions** 

	Homogeneity Property Prediction Based on											
	<ul> <li>Acceptal</li> </ul>	oility Requires a Va	lue > 211 -									
	(Shad	ed Values are Accep	ptable)									
	Target	Measured	Bias-Corrected									
Glass ID	Composition	Composition	Composition									
cst01	197.8	202.5	200.6									
cst02	204.9	206.6	205.8									
cst03	186.2	187.8	186.2									
cst04	205.5	205.1	205.1									
cst05	207.3	207.3 210.2										
cst06	209.7	211.8	209.6									
cst07	203.3	202.2	200.4									
cst08	214.6	216.6	214.2									
cst09	197.5	197.8	197.6									
cst10	208.9	207.9	207.7									
cst11	209.8	207.2	205.3									
cst12	223.3	219.5	219.6									
cst13	206.5	207.6	207.4									
cst14	204.9	203.2	203.3									
cst15	206.5	206.8	206.6									
cst16	204.9	202.4	202.6									
cst17	206.5	208.7	207.1									

The homogeneity constraint was developed for a glass compositional region that does not include all of the components introduced by CST. Therefore, the predictability of phase separation by this model may be questionable. A significant search for amorphous phase separation in these glasses is beyond the scope of work for this task, except when routine SEM analysis is performed.

# XRD and SEM Analysis of the Glasses

None of the quenched glasses exhibited any visually apparent crystalline phases. However, several of the centerline-cooled glasses did have separate phases as observed visually. These glasses were submitted to ADS for X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses. This section presents the results of these analyses.

XRD results were obtained for the following clc glasses:

cst01	Amorphous
cst05	Amorphous
cst06	Hematite and Trevorite and an unidentified phase
cst08	Hematite and Trevorite
cst11	Hematite plus a small amount of an unidentified phase
cst14	Amorphous

Glasses cst01, cst05, and cst14 were amorphous showing no crystalline phases to the sensitivity of XRD (~0.5 to 1 wt% crystals).

Glasses cst06 and cst08 contained Purex sludge at 30 wt% oxide and cst11 contained 28 wt% oxide of the Blend sludge. Therefore, at the higher sludge loadings, it appears that hematite can phase separate. Due to the limited scope for this part of the task, no XRD data was obtained for the other high-sludge-loaded glasses produced under quenched or centerline-cooled conditions.

SEM results were obtained for phases that appear on the surface of the glass. A variety of crystals were observed on the glass surfaces. For cst01, a glass with maximum titanium (~5 wt% oxide), spinels enriched with titanium were evident along with a few rod-like structures of titanium. Glass cst05, with lesser titanium (~3 wt% oxide), showed mainly trevorite with some crystals enriched with titanium.

Glass cst06, with 4 wt% titanium oxide and 30 wt% oxide Purex sludge, exhibited crystals of spinel enriched with titanium, a phase rich in Mn and Fe, and a rod-like phase rich in Fe, Ti, and Si (the Si could result from the matrix). The unidentified phase detected by XRD could correspond to one of the phases observed by SEM.

Glass cst11 has the maximum titanium (5wt% as oxide) concentration in the glass and 28% blend sludge with high Fe concentration. Surface crystals of spinel enriched in titanium were observed. Both rod-like and spots of titanium phases were also observed by SEM. Finally a phase rich in Mn and Fe and similar in signature to the unidentified phase seen in cst06 was observed by SEM. The unidentified phase detected by XRD has similarities, but is evidently not identical, to the unidentified phase in cst06.

For glass cst12, a glass low in titanium (2 wt% as oxide) but high in sludge (30 wt% Blend), the majority of crystals were spinel with a smaller amount of crystals containing the high Mn and Fe phase. One spot was observed to contain higher concentrations of uranium.

For glass cst16, which contains HM sludge, crystals containing Fe, Mn, Ti, Ni, Si and O were observed. Spinel crystals were also detected.

It may also be worth noting that the presence of these crystalline phases had no apparent impact on the durabilities of these glasses.

# **CONCLUSIONS**

This study was conducted to determine the effect, if any, on the PCT responses of glasses cooled at different rates. The glasses contained CST and MST (plus a simulated sludge representing Purex, Blend, or HM) in amounts consistent with coupled operations using the CST option for salt disposition. Two bounding cooling profiles were used in this study: rapidly quenched and canister centerline cooling profiles. Glasses were selected based on a number of criteria, but mainly to challenge the regions where amorphous phase separation might be expected. Currently, DWPF utilizes a homogeneity constraint to preclude glass regions that could lead to phase separation. However, this constraint was not developed for a compositional region that includes the levels of CST (3-9 wt%) and MST (2.5 wt%) introduced by this study. Due to this uncertainty, the CST glasses that were selected for study covered a wide range of values for the homogeneity constraint. Also, it was important to ensure that deleterious phase separation does not occur for either cooling profile. In this case, deleterious phase separation is defined as the formation of amorphous phase separation (or glass-inglass) that significantly alters the glass durability as measured by the PCT response.

The results revealed that there was no practical difference between the PCT responses for glasses subjected to the two cooling profiles. In fact, although to a small extent, the boron PCT responses for the centerline-cooled glasses were more durable than the rapidly quenched glasses. These results reveal that no deleterious phase separation occurred under either cooling regime.

X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to evaluate phases observed in the centerline cooled glasses. XRD analysis, which detects crystals throughout the bulk of the glass, revealed that trevorite and hematite were present in some of the glasses.

SEM analysis of the surface crystals revealed a variety of different species including titanium rods and spots, trevorite, and trevorite enriched with titanium (appears to correlate with the amount of titanium in the glass), and a phase rich in Mn and Fe. One glass, cst12, contained a crystal high in uranium content.

The durabilities, as measured by the PCT response of boron, for the CST glasses ranged from 0.41 to 0.80 g/L. These values are significantly lower than the PCT values for glasses containing PHA (as

reported in WSRC-TR-2001-00123) and for EA. The PCT results are highly clustered consistent with previous work. The measured PCT values fall above the upper 95% prediction interval of the DWPF model used to predict durability, again, consistent with previous results. The very good durability of the CST containing glasses implies that durability may not be the limiting factor for waste loading in this option.

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- [8] Jantzen, C. M., J. B. Pickett, K. G. Brown, T. B. Edwards, and D. C. Beam, "Process/Product Models for the Defense Waste Processing Facility (DWPF): Part I. Predicting Glass Durability from Composition Using a <u>Thermodynamic Hydration Energy Reaction Model</u> (THERMO) (U)," WSRC-TR-93-672, Rev. 1, September 28, 1995.

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# APPENDIX A.

**Supplemental Tables and Exhibits** 

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**Table A.1: Composition of PCT Leachate Solutions** 

				Concentrations in ppm Concentrations in ppm												
Glass	Lab					orted)	111			ng for dilutio	on)	Commo	n Logarithm o	of ppm Concer	trations	
ID	ID	Blk	Seq	В	Li	Na	Si	В	Li	Na	Si	log[B]	log[Li]	log[Na]	log[Si]	
std	std-b1-1	1	1	19.7	9.38	81.4	49.8	19.700	9.380	81.400	49.800	1.2945	0.9722	1.9106	1.6972	
cst01	cp056	1	2	8.53	8.93	18.7	60.9	14.217	14.884	31.167	101.502	1.1528	1.1727	1.4937	2.0065	
cst08	cp022	1	3	9.31	9.52	21.7	62.2	15.517	15.867	36.167	103.669	1.1908	1.2005	1.5583	2.0156	
cst05clc	cp037	1	4	8.94	9.11	22.4	68.1	14.900	15.184	37.334	113.502	1.1732	1.1814	1.5721	2.0550	
cst17clc	cp067	1	5	6.66	7.92	23.4	64.8	11.100	13.200	39.001	108.002	1.0453	1.1206	1.5911	2.0334	
cst11	cp088	1	6	6.55	8.42	7.88	53.9	10.917	14.034	13.134	89.835	1.0381	1.1472	1.1184	1.9534	
cst08clc	cp104	1	7	7.09	7.68	19.3	58.9	11.817	12.800	32.167	98.169	1.0725	1.1072	1.5074	1.9920	
cst03	cp110	1	8	8.03	9.32	20	64	13.384	15.534	33.334	106.669	1.1266	1.1913	1.5229	2.0280	
cst05	cp085	1	9	9.33	10.1	22.5	68.4	15.550	16.834	37.501	114.002	1.1917	1.2262	1.5740	2.0569	
cst10	cp070	1	10	6.72	8.27	10.3	55.2	11.200	13.784	17.167	92.002	1.0492	1.1394	1.2347	1.9638	
std	std-b1-2	1	11	19.4	9.46	83.1	49.2	19.400	9.460	83.100	49.200	1.2878	0.9759	1.9196	1.6920	
blank	cp046	1	12	< 0.150	<0.040	< 0.100	<0.790	0.125	0.033	0.083	0.658 99.835	-0.9031	-1.4771 1.1173	-1.0792	-0.1816	
cst01clc	cp108 cp064	1	13	7.46	7.86	17.4	59.9	12.434 11.050	13.100	29.001		1.0946		1.4624	1.9993	
cst16		1	14 15	6.63	8.2 8.71	23.1 18.1	64.2		13.667	38.501 30.167	107.002 110.169	1.0434	1.1357	1.5855 1.4795	2.0294	
cst03clc cst11clc	cp069 cp059	1	16	8.14 5.58	7.48	8.58	66.1 53.2	13.567 9.300	14.517 12.467	14.300	88.668	1.1325 0.9685	1.1619 1.0958	1.1553	2.0421 1.9478	
cst12	cp039	1	17	7.8	9.19	9.87	61.4	13.000	15.317	16.450	102.335	1.1140	1.1852	1.2162	2.0100	
cst12	cp045	1	18	8.3	9.17	23.2	64.1	13.834	15.284	38.667	106.836	1.1409	1.1842	1.5873	2.0287	
cst12clc	cp055	1	19	6.59	7.43	10.2	54.2	10.984	12.384	17.000	90.335	1.0407	1.0928	1.2305	1.9559	
cst16clc	cp052	1	20	6.49	7.63	22.8	63.4	10.817	12.717	38.001	105.669	1.0341	1.1044	1.5798	2.0239	
cst10clc	cp044	1	21	5.56	7.05	10.3	53.9	9.267	11.750	17.167	89.835	0.9669	1.0700	1.2347	1.9534	
std	std-b1-3	1	22	19.7	9.53	83.3	50.1	19.700	9.530	83.300	50.100	1.2945	0.9791	1.9206	1.6998	
std	std-b2-1	2	1	19.3	9.4	78.4	49.8	19.300	9.400	78.400	49.800	1.2856	0.9731	1.8943	1.6972	
cst03	cp020	2	2	7.48	8.72	15.6	59	12.467	14.534	26.001	98.335	1.0958	1.1624	1.4150	1.9927	
cst12clc	cp097	2	3	7.19	8.28	10.9	59.1	11.984	13.800	18.167	98.502	1.0786	1.1399	1.2593	1.9934	
cst11clc	cp054	2	4	5.25	7.29	8.86	50.7	8.750	12.150	14.767	84.502	0.9420	1.0846	1.1693	1.9269	
cst11	cp084	2	5	6.3	8.24	8.06	51.9	10.500	13.734	13.434	86.502	1.0212	1.1378	1.1282	1.9370	
cst01	cp098	2	6	7.89	8.37	17.3	56.1	13.150	13.950	28.834	93.502	1.1189	1.1446	1.4599	1.9708	
cst17	cp003	2	7	8	9	22.2	62.9	13.334	15.000	37.001	104.835	1.1249	1.1761	1.5682	2.0205	
cst10clc	cp053	2	9	5.26 7.75	6.65 8.07	9.97 21.2	51.6	8.767 12.917	11.084 13.450	16.617 35.334	86.002 102.669	0.9428 1.1112	1.0447 1.1287	1.2206	1.9345 2.0114	
cst17clc cst01clc	cp015 cp030	2	10	1.13	8.07	21.2	61.6	12.917	15.430	33.334	102.009	1.1112	1.1287	1.5482	2.0114	
std	std-b2-2	2	11	19.1	9.34	78	49.5	19.100	9.340	78.000	49.500	1.2810	0.9703	1.8921	1.6946	
cst16clc	cp065	2	12	6.81	8	23.4	66	11.350	13.334	39.001	110.002	1.0550	1.1249	1.5911	2.0414	
cst08clc	cp016	2	13	7.03	7.68	18.9	59	11.717	12.800	31.501	98.335	1.0688	1.1072	1.4983	1.9927	
cst03clc	cp048	2	14	7.73	8.61	17.8	64.2	12.884	14.350	29.667	107.002	1.1100	1.1569	1.4723	2.0294	
cst05	cp109	2	15	8.98	9.68	20.9	66.9	14.967	16.134	34.834	111.502	1.1751	1.2077	1.5420	2.0473	
cst10	cp029	2	16	6.89	8.45	10.5	56.8	11.484	14.084	17.500	94.669	1.0601	1.1487	1.2430	1.9762	
cst08	cp051	2	17	9.37	9.53	21.3	63	15.617	15.884	35.501	105.002	1.1936	1.2010	1.5502	2.0212	
cst12	cp006	2	18	7.8	9.14	9.92	62.5	13.000	15.234	16.534	104.169	1.1140	1.1828	1.2184	2.0177	
cst16	cp105	2	19	6.31	7.85	21.7	60.8	10.517	13.084	36.167	101.335	1.0219	1.1167	1.5583	2.0058	
cst05clc	cp038	2	20	9.5	9.4	22.6	73.1	15.834	15.667	37.667	121.836	1.1996	1.1950	1.5760	2.0858	
std	std-b2-3	2	21	19.4	9.22	77.1	50.1	19.400	9.220	77.100	50.100	1.2878	0.9647	1.8871	1.6998	
std	std-b3-1	3	1	19.7	9.88	78.6	48.9	19.700	9.880	78.600	48.900	1.2945	0.9948	1.8954	1.6893	
cst10 cst05clc	cp095 cp063	3	3	7.31 9.32	8.66 9.66	10.9	56.2 66	12.184 15.534	14.434 16.100	18.167 38.334	93.669 110.002	1.0858 1.1913	1.1594 1.2068	1.2593 1.5836	1.9716 2.0414	
cst05c1c	cp063	3	4	8.33	9.66	18.5	58.5	13.884	15.384	30.834	97.502	1.1913	1.2068	1.3836	1.9890	
cst11clc	cp041	3	5	5.83	8.09	9.32	51.9	9.717	13.484	15.534	86.502	0.9875	1.1298	1.1913	1.9890	
cst12	cp049	3	6	8.13	9.63	10.6	61.4	13.550	16.050	17.667	102.335	1.1319	1.2055	1.2472	2.0100	
cst08	cp043	3	7	9.46	9.62	21.1	61.1	15.767	16.034	35.167	101.835	1.1977	1.2050	1.5461	2.0079	
cst05	cp068	3	8	9.38	10.2	22	66.1	15.634	17.000	36.667	110.169	1.1941	1.2305	1.5643	2.0421	
cst08clc	cp100	3	9	7.63	8.38	19.6	60.2	12.717	13.967	32.667	100.335	1.1044	1.1451	1.5141	2.0015	
cst16clc	cp032	3	10	8.42	8.92	22.5	62.4	14.034	14.867	37.501	104.002	1.1472	1.1722	1.5740	2.0170	
std	std-b3-2	3	11	19.6	9.64	77.2	48.7	19.600	9.640	77.200	48.700	1.2923	0.9841	1.8876	1.6875	
cst01clc	cp080	3	12													
cst10clc	cp062	3	13	6.1	7.49	10.7	55.5	10.167	12.484	17.834	92.502	1.0072	1.0963	1.2512	1.9662	
cst03clc	cp083	3	14	8.69	9.1	17.5	68.6	14.484	15.167	29.167	114.336	1.1609	1.1809	1.4649	2.0582	
cst17clc	cp010	3	15	8.38	8.72	22	61.9	13.967	14.534	36.667	103.169	1.1451	1.1624	1.5643	2.0135	
cst17	cp090	3	16	8.82	9.57	22.4	65.4	14.700	15.950	37.334	109.002	1.1673	1.2028	1.5721	2.0374	
cst12clc	cp099	3	17	8.12	8.93	11.7	63.2	13.534	14.884	19.500	105.335	1.1314	1.1727	1.2900	2.0226	
cst11	cp077	3	18	7.21	8.93	8.56	56.2	12.017	14.884	14.267	93.669	1.0798	1.1727	1.1543	1.9716	
cst03	cp043	3	19	8.93	8.26	16.9 21.5	68.7	14.884 11.334	16.667	28.167	114.502	1.1727	1.2219	1.4497	2.0588	
cst16	cp076 std-b3-3	3	20	6.8	8.26 10.1	79.9	61.6 53	21.000	13.767 10.100	35.834 79.900	102.669 53.000	1.0544 1.3222	1.1388 1.0043	1.5543 1.9025	2.0114 1.7243	
std	Stu-03-3		- 41	- 41	10.1	19.9		41.000	10.100	79.900	23.000	1.3444	1.0043	1.7023	1.7243	

### **Notes:**

- (1). Values below the detection limit (indicated by a "<") were converted to ½ the detection limit.
- (2) The shaded entries indicate that the solution-weight fell outside of the guidelines for a successful PCT result.

 Table A.1: Composition of PCT Leachate Solutions (continued)

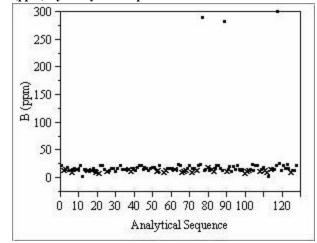
		1	1	Concentrations in ppm				Concentrations in ppm							
Class	Lab						Ш			ng for diluti	on)	Commo	n I aganithm a	of ppm Concer	strations
Glass ID	ID	Blk	Seq	В	(as rep	orted)	Si	В	Li	_	Si				
			_			Na 70.5				Na 70.500		log[B]	log[Li]	log[Na]	log[Si]
std	std-b4-1	4	1	19.3	10.3	79.5	47.9	19.300	10.300	79.500	47.900	1.2856	1.0128	1.9004	1.6803
cst14clc	cp036	4	2	6.66	8.93	19	61.5	11.100	14.884	31.667	102.502	1.0453	1.1727	1.5006	2.0107
cst15	cp008		3	7.64	9.79	16.9	55.3	12.734	16.317	28.167	92.169	1.1050	1.2126	1.4497	1.9646
cst06	cp075	4	4	8.16	9.35	21.2	53.3	13.600	15.584	35.334	88.835	1.1335	1.1927	1.5482	1.9486
cst04clc	cp081	4	5	8.22	8.99	21.6	58.9	13.700	14.984	36.001	98.169	1.1367	1.1756	1.5563	1.9920
cst07clc	cp042	4	6	11.1	11.4	21.7	78.6	18.500	19.000	36.167	131.003	1.2672	1.2788	1.5583	2.1173
cst14	cp087	4	7	6.62	9.41	17.9	61.2	11.034	15.684	29.834	102.002	1.0427	1.1954	1.4747	2.0086
cst09clc	cp057	4	8	8.39	9.27	18.3	59.5	13.984	15.450	30.501	99.169 99.669	1.1456	1.1889	1.4843	1.9964
cst09	cp001		9	8.73	10	17.8	59.8	14.550	16.667	29.667		1.1629	1.2219	1.4723	1.9986
cst15clc	cp103	4	10	8.2	9.54	18.5	62.5	13.667	15.900	30.834	104.169	1.1357	1.2014	1.4890	2.0177
std	std-b4-2	4	11	19.9	10.6	82	49.5	19.900	10.600	82.000	49.500	1.2989	1.0253	1.9138	1.6946
ARM	cp031	4	12	12.9	10	25.5	39.3	21.500	16.667 111.834	42.501	65.501	1.3324	1.2219	1.6284	1.8163
EA	cp047	4	13	17.2	6.71	49.1	30.2	286.667		818.335	503.334	2.4574	2.0486	2.9129	2.7019
cst02clc	cp060	4	14	6.36	9.01	11.1	60.8	10.600	15.017	18.500	101.335	1.0253	1.1766	1.2672	2.0058
cst06clc	cp012	4	15	6.75	8.09	20.3	53.8	11.250	13.484	33.834	89.668	1.0512	1.1298	1.5294	1.9526
cst07	cp086	4	16	11.8	12.8	21.9	79.5	19.667	21.334	36.501	132.503	1.2937	1.3291	1.5623	2.1222
cst02	cp066	4	17	6.02	9.12	9.33	57.5	10.034	15.200	15.550	95.835	1.0015	1.1819	1.1917	1.9815
cst04	cp035	4	18	8.92	9.95	22.5	59.5	14.867	16.584	37.501	99.169	1.1722	1.2197	1.5740	1.9964
cst13	cp039	4	19	7.71	9.63	15.3	57.7	12.850	16.050	25.501	96.169	1.1089	1.2055	1.4065	1.9830
cst13clc	cp089	4	20	7.85	9.35	16.7	60.5	13.084	15.584	27.834	100.835	1.1167	1.1927	1.4446	2.0036
std	std-b4-3	4	21	19.5	10.4	80.1	48.1	19.500	10.400	80.100	48.100	1.2900	1.0170	1.9036	1.6821
std	std-b5-1	5	1	19.2	9.39	81	49.7	19.200	9.390	81.000	49.700	1.2833	0.9727	1.9085	1.6964
cst09	cp034	5	2	8.52	9.57	17.3	61.8	14.200	15.950	28.834	103.002	1.1523	1.2028	1.4599	2.0128
cst14clc	cp072	5	3	5.82	7.68	16.2	61.6	9.700	12.800	27.001	102.669	0.9868	1.1072	1.4314	2.0114
EA	cp014	5	4	16.7	5.71	49	31.3	278.334	95.167	816.668	521.668	2.4446	1.9785	2.9120	2.7174
cst13	cp073	5	5	7.05	8.72	12.6	58.6	11.750	14.534	21.000	97.669	1.0700	1.1624	1.3222	1.9898
cst13clc	cp028	5	6	6.92	8.16	14	59.6	11.534	13.600	23.334	99.335	1.0620	1.1335	1.3680	1.9971
cst07clc	cp027	5	7	9.97	10.3	18.9	74.7	16.617	17.167	31.501	124.503	1.2206	1.2347	1.4983	2.0952
cst02	cp040	5	8	5.22	8.13	6.18	57.8	8.700	13.550	10.300	96.335	0.9395	1.1319	1.0128	1.9838
cst07	cp025	5	9	10.4	10.9	18.1	74.6	17.334	18.167	30.167	124.336	1.2389	1.2593	1.4795	2.0946
cst15clc	cp093	5	10	6.83	7.87	14.8	58.2	11.384	13.117	24.667	97.002	1.0563	1.1178	1.3921	1.9868
std	std-b5-2	5	11	19.4	9.58	82.1	50.5	19.400	9.580	82.100	50.500	1.2878	0.9814	1.9143	1.7033
cst15	cp074	5	12	7.42	8.82	14.3	60.8	12.367	14.700	23.834	101.335	1.0923	1.1673	1.3772	2.0058
cst14	cp101	5	13	5.91	8.08	14.4	62.7	9.850	13.467	24.000	104.502	0.9934	1.1293	1.3802	2.0191
cst06	cp061	5	14	7.43	8.13	18.4	54.3	12.384	13.550	30.667	90.502	1.0928	1.1319	1.4867	1.9567
cst02clc	cp050	5	15	5.24	7.31	7.02	58.6	8.734	12.184	11.700	97.669	0.9412	1.0858	1.0682	1.9898
cst09clc	cp078	5	16	7.45	7.86	14.8	57.7	12.417	13.100	24.667	96.169	1.0940	1.1173	1.3921	1.9830
cst04	cp058	5	17	8.19	8.59	19.1	60.3	13.650	14.317	31.834	100.502	1.1351	1.1559	1.5029	2.0022
cst04clc	cp013	5	18	7.53	7.9	18.8	59.1	12.550	13.167	31.334	98.502	1.0987	1.1195	1.4960	1.9934
ARM	cp102	5	19	12.2	8.91	23.2	40.1	20.334	14.850	38.667	66.835	1.3082	1.1717	1.5873	1.8250
cst06clc	cp023	5	20	5.87	6.7	16.5	53.2	9.784	11.167	27.501	88.668	0.9905	1.0479	1.4393	1.9478
std	std-b5-3	5	21	19.2	9.46	81	50	19.200	9.460	81.000	50.000	1.2833	0.9759	1.9085	1.6990
std	std-b6-1	6	1	20	10.3	81.9	51	20.000	10.300	81.900	51.000	1.3010	1.0128	1.9133	1.7076
cst13clc	cp024	6	2	7.57	8.97	16.5	62	12.617	14.950	27.501	103.335	1.1010	1.1747	1.4393	2.0142
cst15clc	cp002	6	3	7.62	8.83	17.7	61.9	12.700	14.717	29.501	103.169	1.1038	1.1678	1.4698	2.0135
cst09	cp005	6	4	9.02	10	18.1	65	15.034	16.667	30.167	108.336	1.1771	1.2219	1.4795	2.0348
cst06	cp026	6	5	7.87	8.92	20.6	54.8	13.117	14.867	34.334	91.335	1.1178	1.1722	1.5357	1.9606
cst14clc	cp094	6	6	6.62	8.82	18.5	67.1	11.034	14.700	30.834	111.836	1.0427	1.1673	1.4890	2.0486
blank	cp071	6	7	< 0.150	<0.040	< 0.100	<0.790	0.125	0.033	0.083	0.658	-0.9031	-1.4771	-1.0792	-0.1816
cst04	cp021	6	8	8.88	9.62	22.1	63.7	14.800	16.034	36.834	106.169	1.1703	1.2050	1.5663	2.0260
cst09clc	cp107	6	9	7.92	8.66	17.7	60	13.200	14.434	29.501	100.002	1.1206	1.1594	1.4698	2.0000
cst13	cp019	6	10	7.45	9.23	14.7	61.1	12.417	15.384	24.500	101.835	1.0940	1.1871	1.3892	2.0079
std	std-b6-2	6	11	20	10.1	80.8	51.2	20.000	10.100	80.800	51.200	1.3010	1.0043	1.9074	1.7093
EA	cp092	6	12	17.8	6.34	50	32.4	296.667	105.667	833.335	540.001	2.4723	2.0239	2.9208	2.7324
ARM	cp007	6	13	13.2	9.67	25.2	42.1	22.000	16.117	42.001	70.168	1.3424	1.2073	1.6233	1.8461
cst06clc	cp018	6	14	6.39	7.61	19.3	55.5	10.650	12.684	32.167	92.502	1.0274	1.1032	1.5074	1.9662
cst07clc	cp033	6	15	11.2	11.2	21.4	81.7	18.667	18.667	35.667	136.169	1.2711	1.2711	1.5523	2.1341
cst15	cp079	6	16	8.1	9.68	16.9	65.1	13.500	16.134	28.167	108.502	1.1303	1.2077	1.4497	2.0354
cst07	cp011	6	17	12.4	12.6	21.7	86.9	20.667	21.000	36.167	144.836	1.3153	1.3222	1.5583	2.1609
cst04clc	cp106	6	18	8.29	8.86	21.6	63.4	13.817	14.767	36.001	105.669	1.1404	1.1693	1.5563	2.0239
cst02clc	cp017	6	19	5.74	8.14	9.75	61.3	9.567	13.567	16.250	102.169	0.9808	1.1325	1.2109	2.0093
cst02	cp004	6	20	6.06	9.1	9.33	63.6	10.100	15.167	15.550	106.002	1.0043	1.1809	1.1917	2.0253
cst14	cp096	6	21	6.36	8.86	17.3	64.4	10.600	14.767	28.834		1.0253	1.1693	1.4599	2.0307
std	std-b6-3	6	22	20.1	9.99	79.3	51.3	20.100	9.990	79.300	51.300	1.3032	0.9996	1.8993	1.7101

### **Notes:**

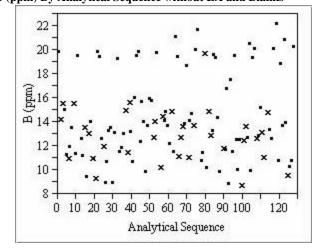
- (1). Values below the detection limit (indicated by a "<") were converted to ½ the detection limit.
- (3) The shaded entries indicate that the solution-weight fell outside of the guidelines for a successful PCT result.

Exhibit A.1: Plots of the Leachate Concentrations in Analytical Sequence by Element

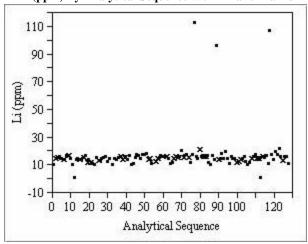
B (ppm) By Analytical Sequence with EA and Blanks



B (ppm) By Analytical Sequence without EA and Blanks



Li (ppm) By Analytical Sequence with EA and Blanks



Li (ppm) By Analytical Sequence without EA and Blanks

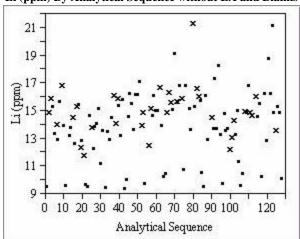
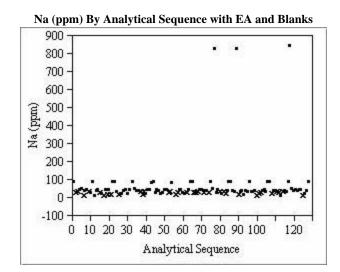
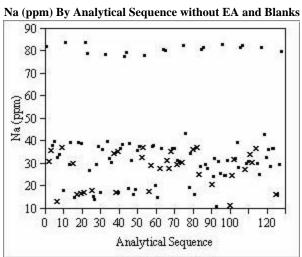
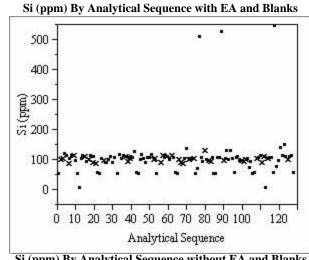


Exhibit A.1: Plots of the Leachate Concentrations in Analytical Sequence by Element







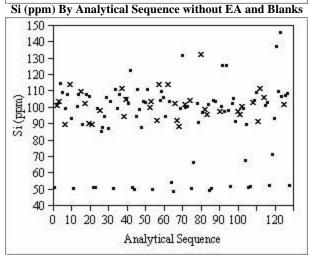
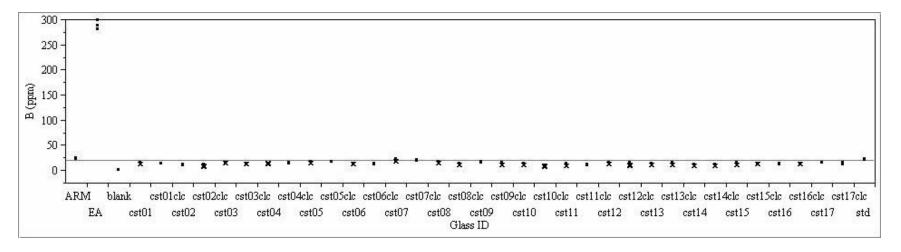


Exhibit A.2: Plots of the Leachate Concentrations by Glass ID by Element

# **B** (ppm) By Sample ID



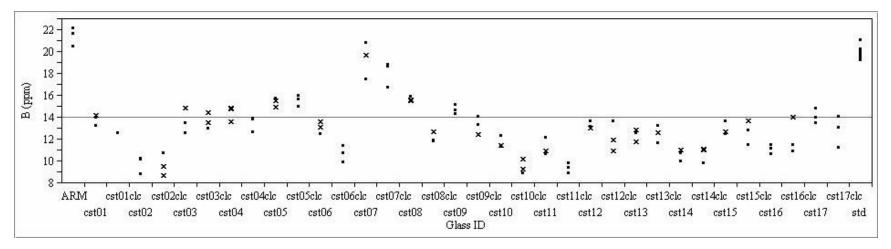
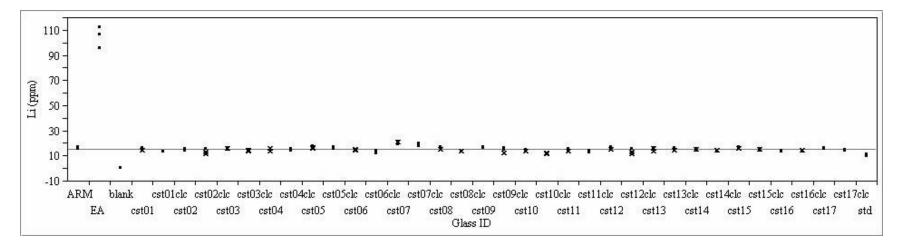


Exhibit A.9: Plots of the Leachate Concentrations by Glass ID by Element (continued)

# Li (ppm) By Sample ID



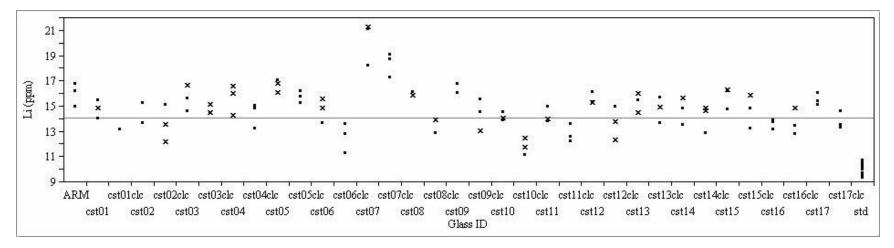
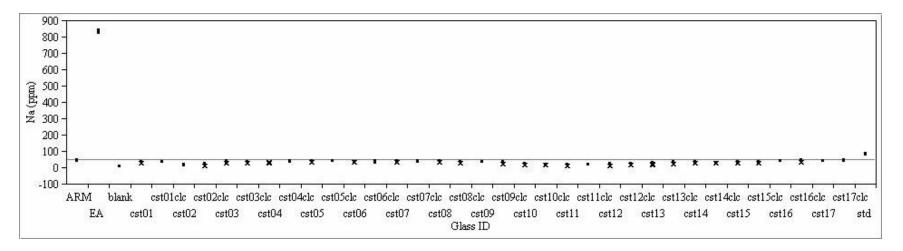


Exhibit A.2: Plots of the Leachate Concentrations by Glass ID by Element (continued)

# Na (ppm) By Sample ID



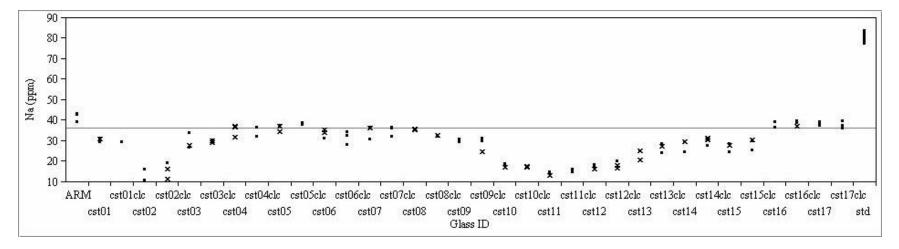
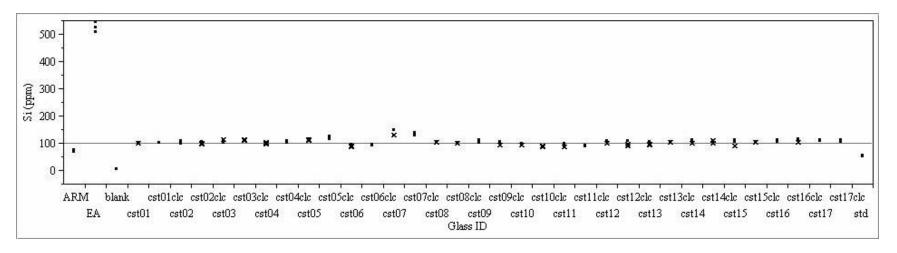


Exhibit A.2: Plots of the Leachate Concentrations by Glass ID by Element (continued)

# Si (ppm) By Sample ID



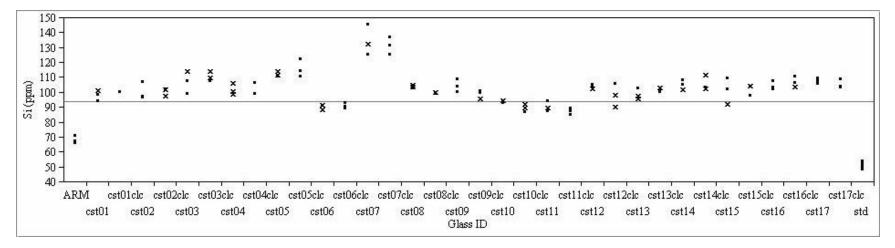
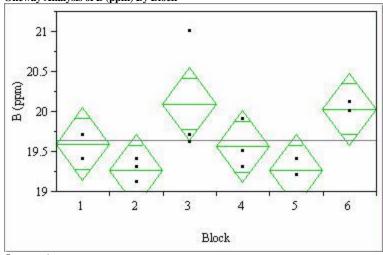


Exhibit A.3: Plots of the Multi-Element Solution Standard by Element





# Oneway Anova

Summary	of	Fit

 Rsquare
 0.558446

 Adj Rsquare
 0.374465

 Root Mean Square Error
 0.359011

 Mean of Response
 19.63889

 Observations (or Sum Wgts)
 18

#### **Analysis of Variance**

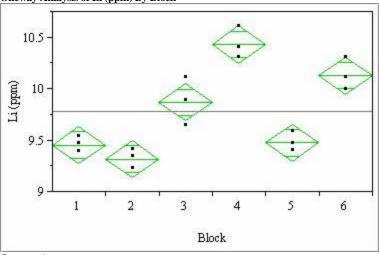
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	1.9561111	0.391222	3.0353	0.0534
Error	12	1.5466667	0.128889		
C. Total	17	3.5027778			

#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	19.6000	0.20728	19.148	20.052
2	3	19.2667	0.20728	18.815	19.718
3	3	20.1000	0.20728	19.648	20.552
4	3	19.5667	0.20728	19.115	20.018
5	3	19.2667	0.20728	18.815	19.718
6	3	20.0333	0.20728	19.582	20.485

Std Error uses a pooled estimate of error variance

#### Oneway Analysis of Li (ppm) By Block



#### **Oneway Anova**

#### **Summary of Fit**

Rsquare	0.921048
Adj Rsquare	0.888151
Root Mean Square Error	0.143894
Mean of Response	9.781667
Observations (or Sum Wgts)	18

#### **Analysis of Variance**

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	2.8985833	0.579717	27.9981	<.0001
Error	12	0.2484667	0.020706		
C Total	17	3 1470500			

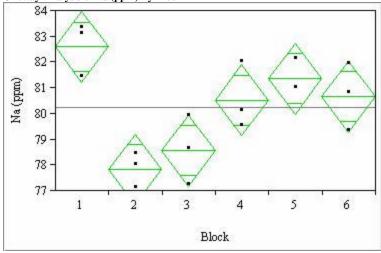
#### Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	9.4567	0.08308	9.276	9.638
2	3	9.3200	0.08308	9.139	9.501
3	3	9.8733	0.08308	9.692	10.054
4	3	10.4333	0.08308	10.252	10.614
5	3	9.4767	0.08308	9.296	9.658
6	3	10.1300	0.08308	9.949	10.311

Std Error uses a pooled estimate of error variance

Exhibit A.3: Plots of the Multi-Element Solution Standard by Element (continued)

Oneway Analysis of Na (ppm) By Block



# Oneway Anova

**Summary of Fit** 

 Rsquare
 0.766645

 Adj Rsquare
 0.669413

 Root Mean Square Error
 1.092906

 Mean of Response
 80.26111

 Observations (or Sum Wgts)
 18

**Analysis of Variance** 

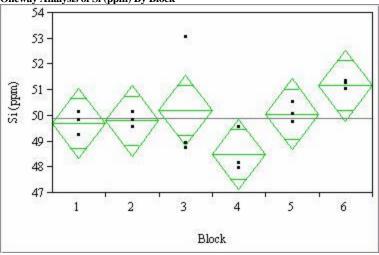
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	47.089444	9.41789	7.8847	0.0017
Error	12	14.333333	1.19444		
C. Total	17	61.422778			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	82.6000	0.63099	81.225	83.975
2	3	77.8333	0.63099	76.459	79.208
3	3	78.5667	0.63099	77.192	79.941
4	3	80.5333	0.63099	79.159	81.908
5	3	81.3667	0.63099	79.992	82.741
6	3	80.6667	0.63099	79.292	82.041

Std Error uses a pooled estimate of error variance

Oneway Analysis of Si (ppm) By Block



#### Oneway Anova

**Summary of Fit** 

 Rsquare
 0.43959

 Adj Rsquare
 0.206086

 Root Mean Square Error
 1.090617

 Mean of Response
 49.90556

 Observations (or Sum Wgts)
 18

**Analysis of Variance** 

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	11.196111	2.23922	1.8826	0.1713
Error	12	14.273333	1.18944		
C Total	17	25 460444			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	49.7000	0.62967	48.328	51.072
2	3	49.8000	0.62967	48.428	51.172
3	3	50.2000	0.62967	48.828	51.572
4	3	48.5000	0.62967	47.128	49.872
5	3	50.0667	0.62967	48.695	51.439
6	3	51.1667	0.62967	49.795	52.539

Std Error uses a pooled estimate of error variance

Exhibit A.4: Correlations and Scatter Plots of the Normalized PCT's before Screening for Solution Weight Loss Problems

## **Measured Compositions – Linear Correlations & Scatter Plots**

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.8015	0.7572	0.7496
log NL[Li (g/L)]	0.8015	1.0000	0.3791	0.5394
log NL[Na (g/L)]	0.7572	0.3791	1.0000	0.7194
log NL[Si (g/L)]	0.7496	0.5394	0.7194	1.0000

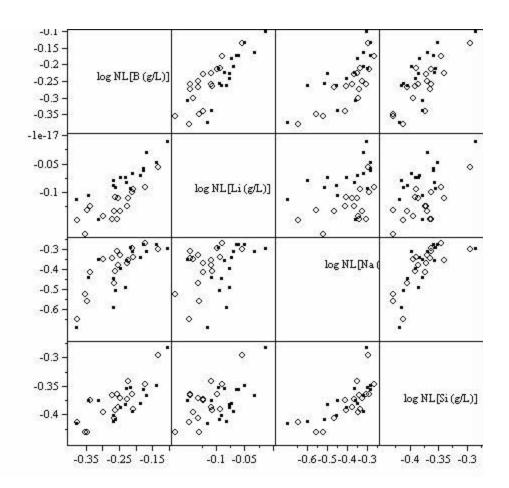


Exhibit A.4: Correlations and Scatter Plots of the Normalized PCT's before Screening for Solution Weight Loss Problems (continued)

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (G/L)]
log NL[B (g/L)]	1.0000	0.7982	0.7610	0.7459
log NL[Li (g/L)]	0.7982	1.0000	0.3810	0.5303
log NL[Na (g/L)]	0.7610	0.3810	1.0000	0.7165
log NL[Si (G/L)]	0.7459	0.5303	0.7165	1.0000

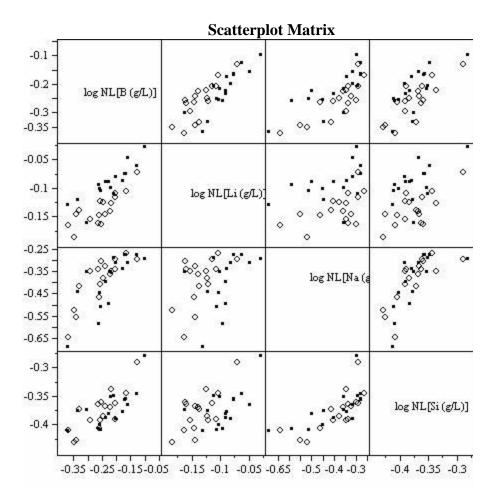


Exhibit A.4: Correlations and Scatter Plots of the Normalized PCT's before Screening for Solution Weight Loss Problems (continued)

**Target Compositions – Linear Correlations & Scatter Plots** 

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.8116	0.7823	0.7935
log NL[Li (g/L)]	0.8116	1.0000	0.4185	0.5723
log NL[Na (g/L)]	0.7823	0.4185	1.0000	0.8280
log NL[Si (g/L)]	0.7935	0.5723	0.8280	1.0000

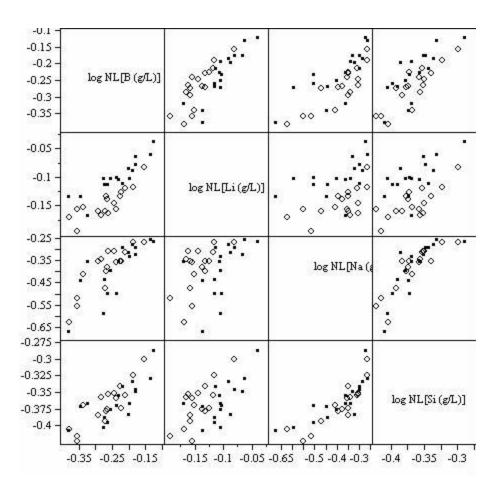


Exhibit A.5: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems

Measured Compositions

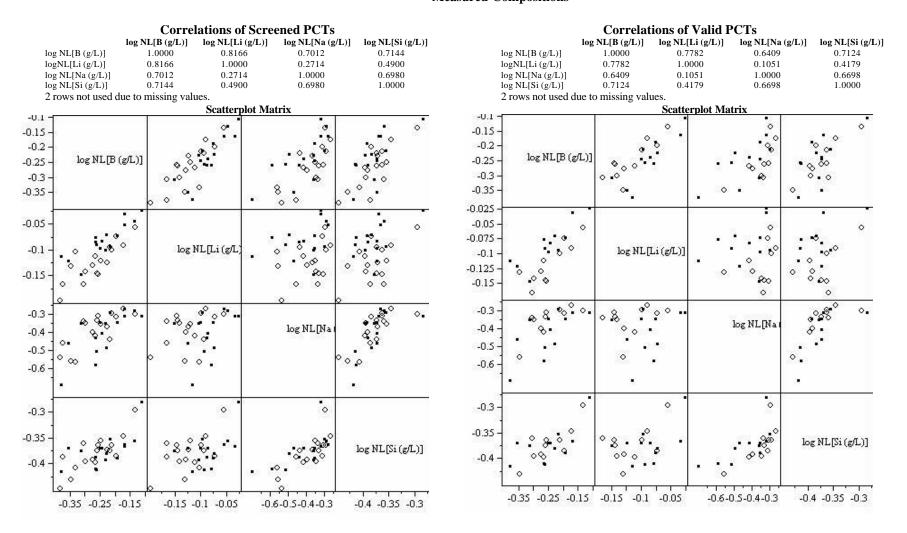


Exhibit A.5: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems (continued)

Measured Bias-Corrected Compositions

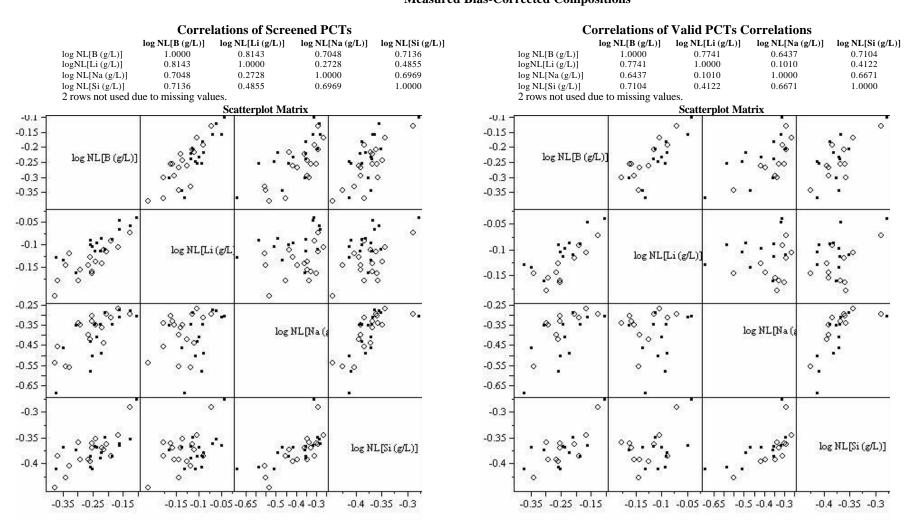


Exhibit A.5: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems (continued)

Targeted Compositions

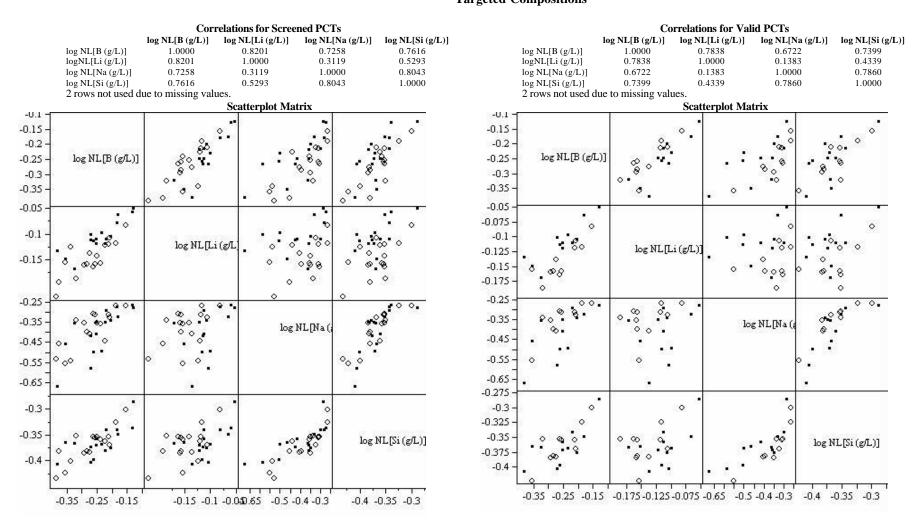
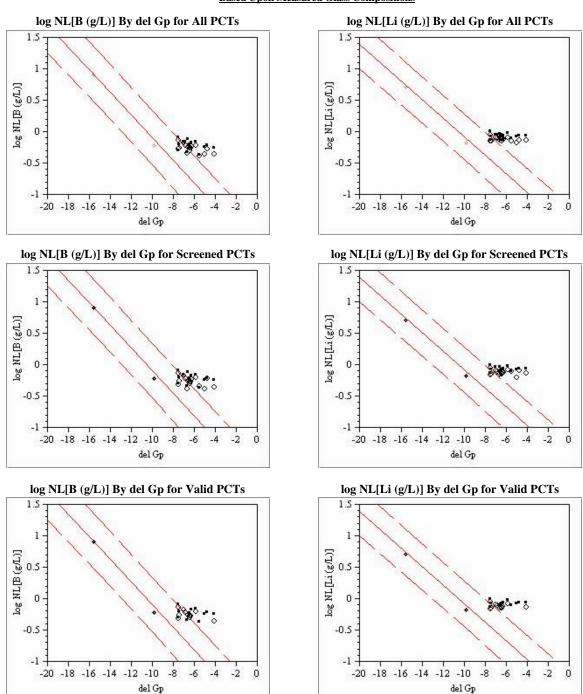


Exhibit A.6: Durability Model Predictions versus PCT Results

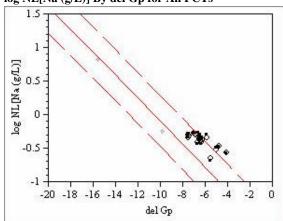
<u>Based Upon Measured Glass Compositions</u>



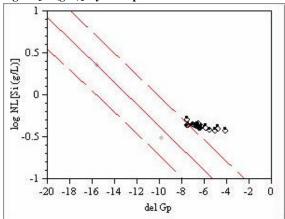
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Based Upon Measured Glass Compositions (continued)

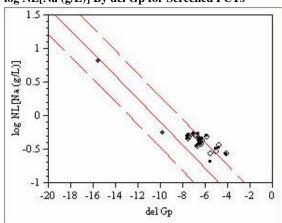
### log NL[Na (g/L)] By del Gp for All PCTs



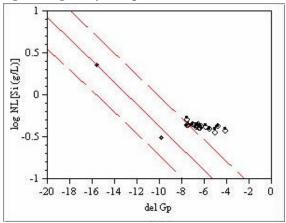
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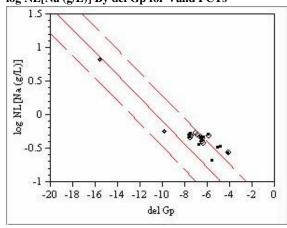
log NL[Na (g/L)] By del Gp for Screened PCTs



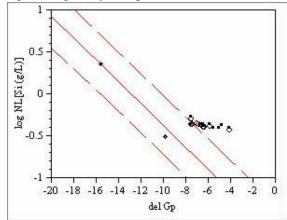
log NL[Si (g/L)] By del Gp for Screened PCTs



log NL[Na (g/L)] By del Gp for Valid PCTs



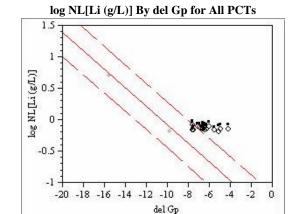
log NL[Si (g/L)] By del Gp for Valid PCTs



 $\underline{\textbf{Based Upon Bias-Corrected, Measured Glass Compositions}}(continued)$ 



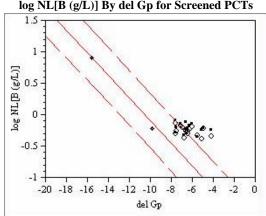
log NL[B(g/L)] -0.5



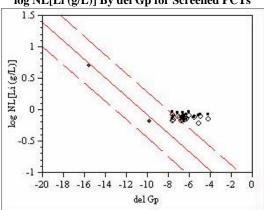
log NL[B (g/L)] By del Gp for Screened PCTs

-10 del Gp

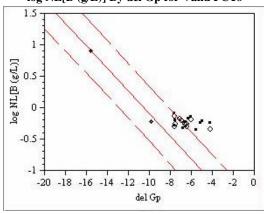
-16 -14 -12



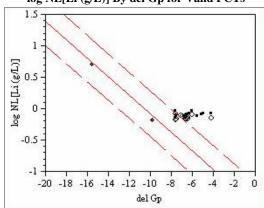




log NL[B (g/L)] By del Gp for Valid PCTs

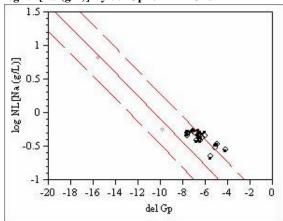


 $log\;NL[Li\;(g/L)]\;By\;del\;Gp\;for\;Valid\;PCTs$ 

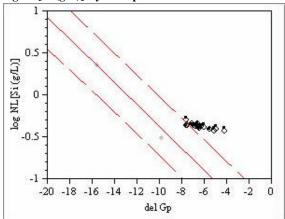


 $\underline{\textbf{Based Upon Bias-Corrected, Measured Glass Compositions}}(continued)$ 

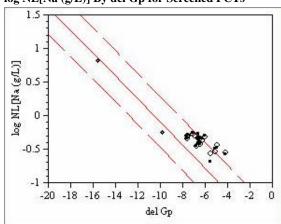
### log NL[Na (g/L)] By del Gp for All PCTs



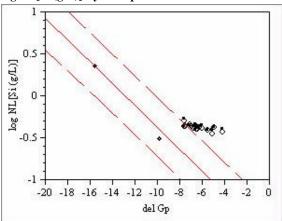
log NL[Si (g/L)] By del Gp for All PCTs



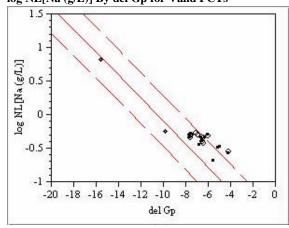
log NL[Na (g/L)] By del Gp for Screened PCTs



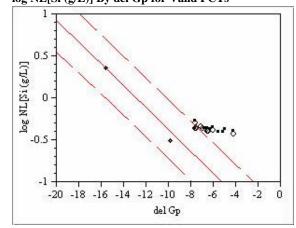
log NL[Si (g/L)] By del Gp for Screened PCTs



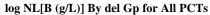
log NL[Na (g/L)] By del Gp for Valid PCTs

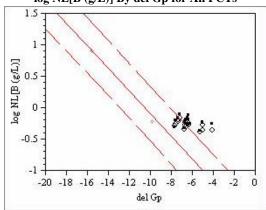


log NL[Si (g/L)] By del Gp for Valid PCTs

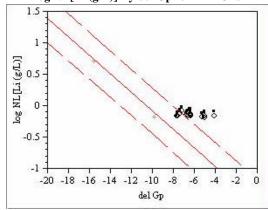


Based Upon Targeted Glass Compositions(continued)

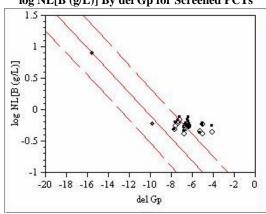




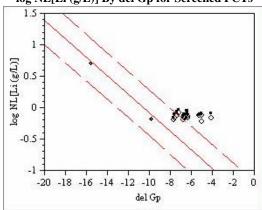
### log NL[Li (g/L)] By del Gp for All PCTs



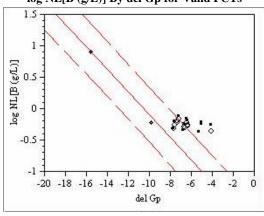
log NL[B (g/L)] By del Gp for Screened PCTs



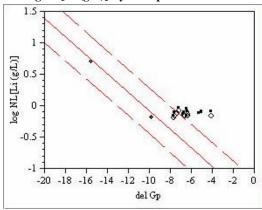
log NL[Li (g/L)] By del Gp for Screened PCTs



log NL[B (g/L)] By del Gp for Valid PCTs



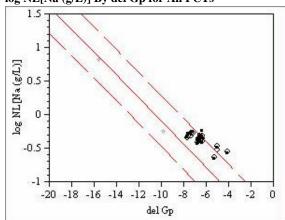
log NL[Li (g/L)] By del Gp for Valid PCTs



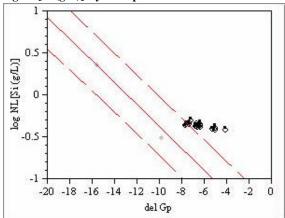
37

Based Upon Targeted Glass Compositions(continued)

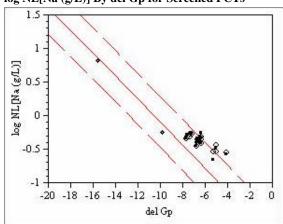
### log NL[Na (g/L)] By del Gp for All PCTs



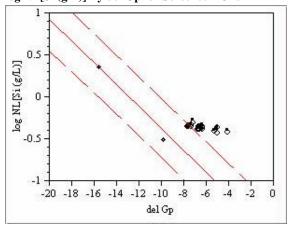
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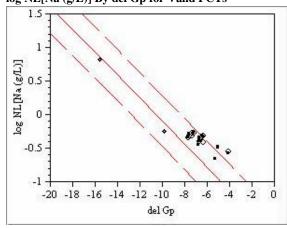
log NL[Na (g/L)] By del Gp for Screened PCTs



log NL[Si (g/L)] By del Gp for Screened PCTs



log NL[Na (g/L)] By del Gp for Valid PCTs



log NL[Si (g/L)] By del Gp for Valid PCTs

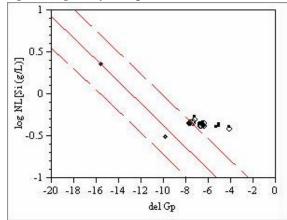
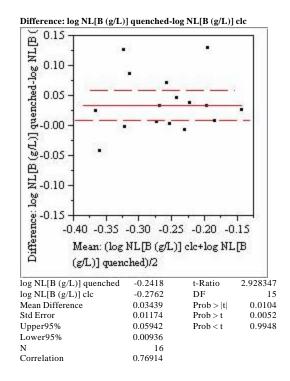


Exhibit A.7: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations, Using Only Those Screened For Water Loss Problems, and Using Only Valid PCTs

#### Difference: log NL[B (g/L)] quenched-log NL[B (g/L)] clc Difference: log NL[B (g/L)] quenched-log NL[B -0.40 -0.35 -0.30 -0.25 -0.20 -0.15 -0.10 Mean: (log NL[B (g/L)] clc+log NL[B (g/L)] quenched)/2 log NL[B (g/L)] quenched -0.2356 3.311563 t-Ratio log NL[B (g/L)] clc -0.2691DF 16 0.0044 Mean Difference 0.03346 Prob > |t|Std Error 0.01011 0.0022 Prob > tUpper95% 0.05489 Prob < t0.9978 Lower95% 0.01204 17 0.80884 Correlation

# Only Results for PCTs Passing Water Loss (i.e. Screened)



### **Valid PCTs Only**

(i.e., those with at least 2 of the triplicates OK).

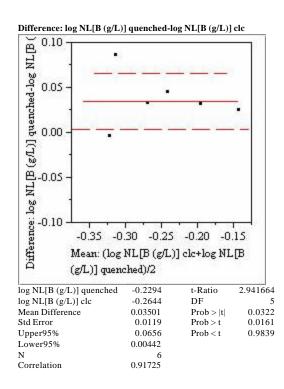
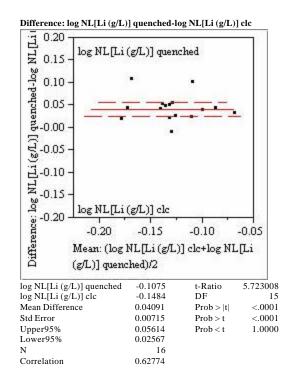


Exhibit A.7: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems

(continued)

#### Difference: log NL[Li (g/L)] quenched-log NL[Li (g/L)] clc 0.10 Difference: log NL[Li(g/L)] quenched-log NL[Li 0.05 0.00 -0:05 -0.10 -0.05 -0.20-0.15 Mean: (log NL[Li (g/L)] clc+log NL[Li (g/L)] quenched)/2 log NL[Li (g/L)] quenched -0.1019 7.639044 t-Ratio log NL[Li (g/L)] clc -0.1449 DF 16 Mean Difference 0.04294 Prob > |t|<.0001 Std Error 0.00562 Prob > t<.0001 Upper95% 0.05485 Prob < t1.0000 Lower95% 0.03102 Ν 17 Correlation 0.67427

# Only Results for PCTs Passing Water Loss (i.e. Screened)



## Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).

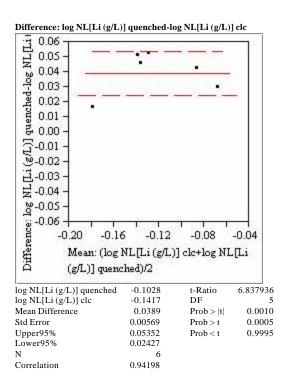
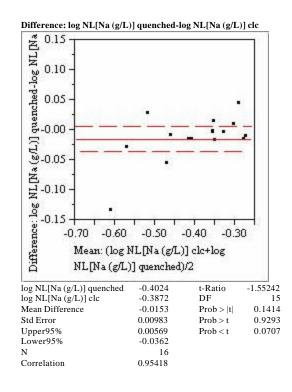


Exhibit A.7: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems
(continued)

#### Difference: log NL[Na (g/L)] quenched-log NL[Na (g/L)] clc NLINA 0.05 0.04 gor-ped-log 0.03 0.02 0.01 0.00 Difference: log NL[Na(g/L)] -0.01 -0.02 -0.03 -0.04 -0.05 -0.50 -0.70 -0.60 -0.40 -0.30 Mean: (log NL[Na (g/L)] clc+log NL[Na (g/L)] quenched)/2 -1.07103 log NL[Na (g/L)] quenched -0.3933 t-Ratio log NL[Na (g/L)] clc -0.386 DF 16 Prob > |t|Mean Difference -0.0073 0.3000 Std Error 0.00685 Prob > t0.8500 Upper95% 0.00718 Prob < t0.1500 Lower95% -0.0218 Ν 17 Correlation 0.97997

# Only Results for PCTs Passing Water Loss (i.e. Screened)



Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).

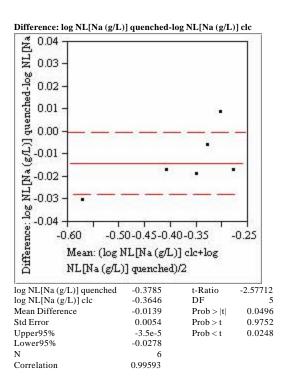
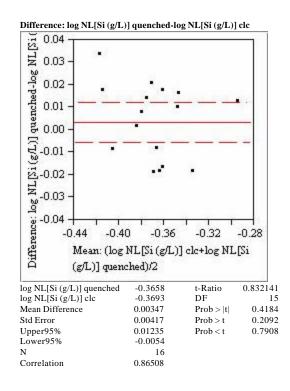


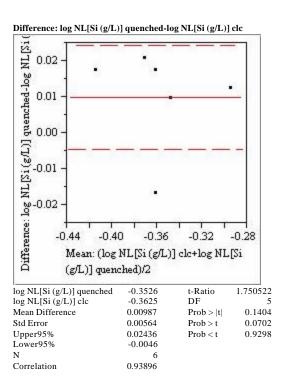
Exhibit A.7: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems
(continued)

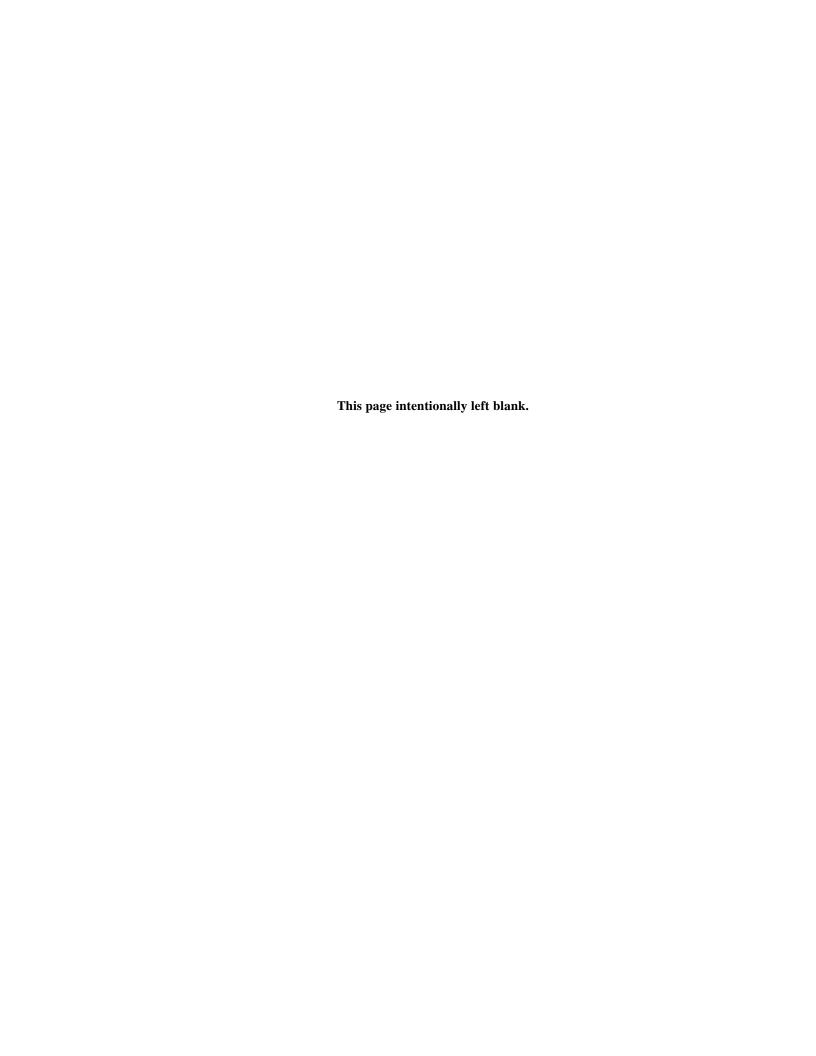
#### Difference: log NL[Si (g/L)] quenched-log NL[Si (g/L)] clc -0.40 -0.36 -0.32-0.28-0.44 Mean: (log NL[Si (g/L)] clc+log NL[Si (g/L)] quenched)/2 0.872787 log NL[Si (g/L)] quenched -0.3648 t-Ratio log NL[Si (g/L)] clc -0.3677 DF 16 Mean Difference 0.00288 Prob > |t|0.3957 Std Error 0.00329 Prob > t0.1978 Upper95% 0.00986 Prob < t0.8022 Lower95% -0.0041Ν 17 Correlation 0.90394

# Only Results for PCTs Passing Water Loss (i.e. Screened)



## Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).





### WSRC-TR-2001-00125 Revision 0

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- TIM (4 copies), 703-43A

### DOE-SR

J. W. McCullough, Jr., 704-3N

J. M. Reynolds, II, 704-S

P. C. Suggs, 704-196N

### **Pacific Northwest National Laboratory**

PO Box 999, MSIN Richland, WA 99352

Attn: D. W. Wester, P7-25